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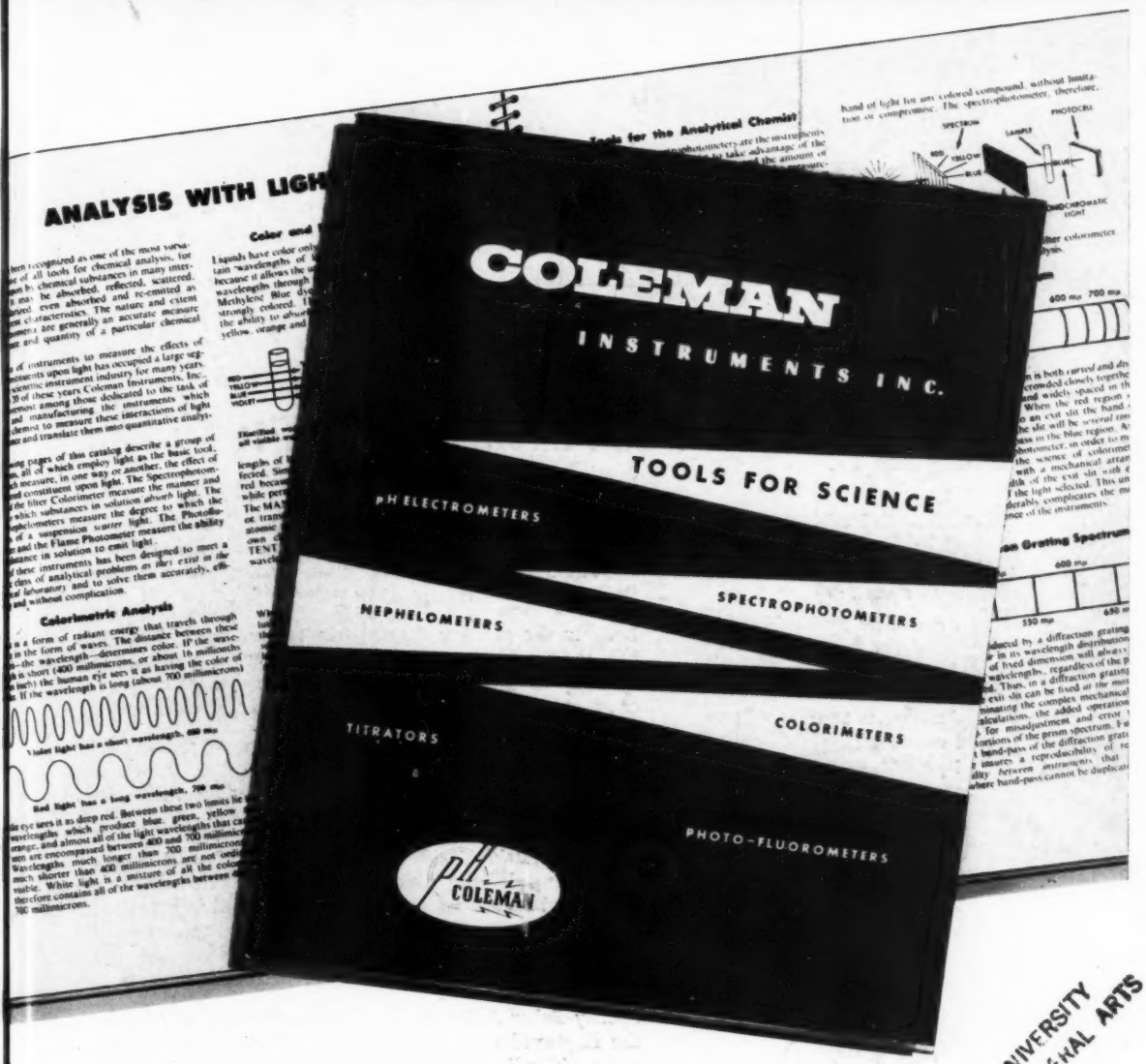

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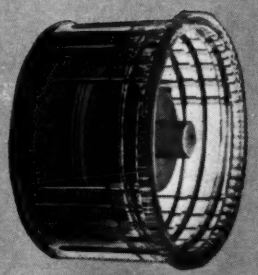

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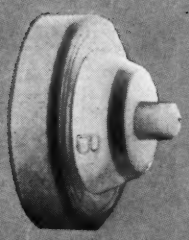

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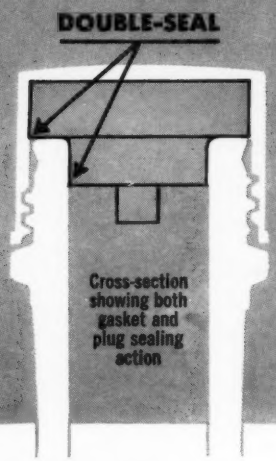

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Subscription Price: Domestic $3.50 per year; Countries in Pan-American Union $3.50 per year; Canada $4.00; other countries $4.50. Issued monthly. Single copies of issues in current year, 50¢ each; all other single copies, one dollar. Foreign remittances must be accompanied by International Money Order.

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The JOURNAL OF CHEMICAL EDUCATION is the official organ of the Divisions of Chemical Education and History of Chemistry and incorporates the Report of the New England Association of Chemistry Teachers, and the Proceedings of the Pacific Southwest Association of Chemistry Teachers. It is indexed in the Education Index, Industrial Arts Index, and, in part, in the Quarterly Cumulative Index Medicus.

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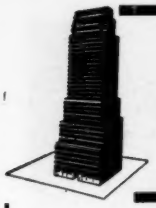

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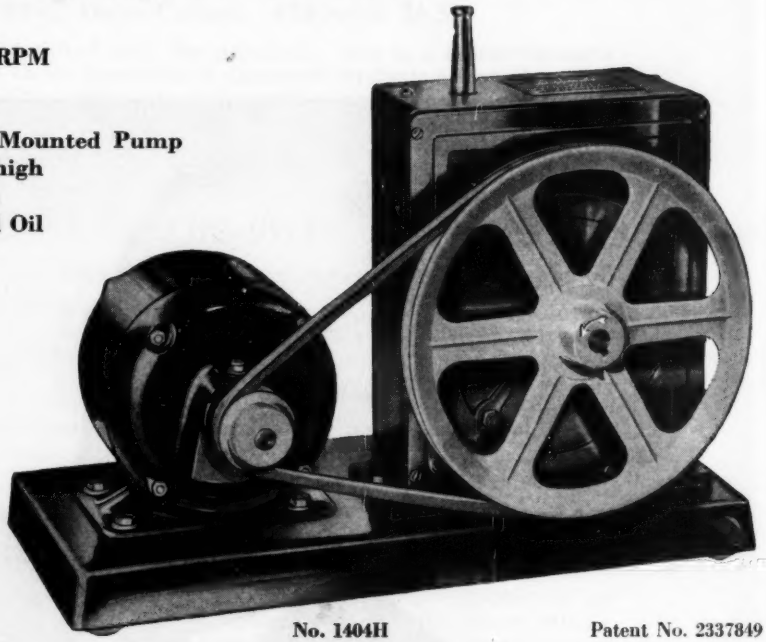

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Editor's Outlook

THE training of physical scientists in the United States can be, very possibly, as vitally important to the defense of this country as the absence of communists in the federal government—Mr. McCarthy to the contrary notwithstanding.

A recent article under this title[1] raises some very interesting questions and makes some very interesting, and unorthodox, proposals. The author, Marshall J. Walker, of the University of Connecticut, is a young man with a very intense enthusiasm for the value of mathematics and the physical sciences, not only for the future welfare of the country but also for the general education of any and everyone. He makes the strongest attack I have yet seen on the current tendency in our secondary schools to slight these subjects in favor of easier and momentarily more popular ones.

To be sure, he does not subscribe to the view that I expressed here last month, that American scientists are the equal of any in the world; indeed, he quotes some rather telling evidence to the contrary. He finds our higher educational system seriously substandard in comparison with the English, although Campbell's comparison, which we published last month, does not altogether bear this out. Nevertheless, Walker says, American Rhodes Scholars have always found it difficult to compete with their younger English classmates at Oxford, and their records have been almost hopelessly behind those of Rhodes Scholars from the Universities of Australia and New Zealand.

But this is not the main point; we must deal with the condition in our schools *today*, and we can agree that among the most dangerous elements in our secondary school system is, first, the increasing tendency to discriminate against mathematics and the physical sciences, and second, our disregard for the small but important percentage of really superior students, in the effort to satisfy the limited educational needs of everybody.

It may be, as the author thinks, that our situation is deteriorating. "The scientists of the United States today include many who were trained in European schools. The remainder are the product of the United States schools of thirty years ago, when the educational accent was still on preparation for college."

His main thesis, however, which leads to his later proposal, is expressed:

> The most important single reason for the inferiority of our graduates in science is that mathematical preparation for serious study is not given early enough. No quantitative science can be taught in an efficient way until the student has mastered calculus. Currently, most students in the United States complete elementary calculus in their fourteenth year. The technical student in a European secondary school completes the same preparation in the twelfth year. This two-year delay is a fatal handicap and by itself is sufficiently serious to account for the poor showing of the United States among the Nobel Prize winners in the physical sciences. In physics and chemistry, a man's peak production occurs near thirty years of age. The typical European student receives his university degree at the age of twenty-four; the typical student in the United States receives his Ph.D. at the age of twenty-seven. This leaves the researcher of the United States with a productive "half-life" only half as long as his foreign competitor's.

But his proposal for action is the real feature, and it intrigues one more as he thinks about its possibilities:

> In a typical high school about 7 per cent of the students will have sufficient ability to pursue graduate work. This 7 per cent is to be provided with a special instructor who will help them learn mathematics, physics, and chemistry. In the beginning, their remaining subjects—English, history, languages, and so on—would be learned from the regular teachers following the regular schedule. The four-year course in mathematics could be divided somewhat as follows: first year, geometry and trigonometry; second year, algebra; third year, analytic geometry and differential calculus; and fourth year, integral calculus and ordinary differential equations. The physics and chemistry courses would differ from the present ordinary courses not so much in content, but chiefly in the additional depth provided by an instructor thoroughly acquainted with the field. . . . .
>
> The teacher of such a course should have a Ph.D. with his major and two minors chosen as any combination of physics, chemistry, and mathematics. The type of man desired will not be tempted by industrial salaries, but he should be paid at least the salary given an assistant professor of these subjects in the state university. For the man who wants to teach, this aggressive 7 per cent of students will offer an opportunity which he would seldom encounter undiluted in any ordinary college. It is suggested that these instructors be employees of the appropriate science of mathematics department of the state university. Their services would be available to any high school which could provide proper space and equipment, and a guaranty that the class would number not more than twenty students.

This is a new type of "university extension" which I must say appeals to me. It is an attractive solution to the question of what to do about the superior students, who now seem to be little more than a nuisance to many high-school administrations.

The author neatly and firmly disposes of some possible objections: Won't this proposal cost a lot of money? (Perhaps, so what?) Isn't this undemocratic? (Quite the contrary, any student has a chance.) Are there enough high-school students who could follow such a program? (Certainly!) Is it wise to encourage specialization so early? (But this isn't specialization.)

A strong point is made of the value of this procedure in general education, whether or not there is any "carry over" of training into other fields. Such a course as that proposed is worth while for its own sake, if not as preparation for future research. His "punch line" is: "Mathematics courses through calculus are the 'children's diseases' of learning, and one who has not had them cannot consider himself completely adult."

Even if this proposal shouldn't prevent the drift away from the exact sciences, it might help to keep them where they will do the most good.

[1] WALKER, MARSHALL J., *J. of Higher Educ.*, **8**, 417 (1954).

THE EVOLUTION OF VALENCE THEORY AND BOND SYMBOLISM

HENRY MACKLE
The Queen's University, Belfast, Northern Ireland

THROUGHOUT the historical literature of chemistry there are scattered fragmentary accounts of various phases in the development of the theory of chemical valence. Often, however, these are merely incidental to some other theme, chiefly that of atomism. It appeared worth while, therefore, to present, primarily for its own sake, the evolution of valence theory from its earliest beginnings up to the advent of the electronic interpretation. Such is the essential aim of what follows. It should not be judged as an excursion into the field of detailed historical scholarship; that would be a very major undertaking for such a subject. It is an attempt, rather, to collect into one place the more important facts and opinions, and to give them such perspective as may be of interest to the chemist as distinct from the historian or the philosopher. When possible the primary sources have been consulted, but sometimes the writer has had to rely on the secondary ones of recognized historians of science.

The earliest published notions on the discreteness of matter include attempts to describe how the ultimate particles are held together. About 430 B.C. Leucippus interpreted chemical combination in terms of an "entanglement" of atoms. His ideas are presented in some detail by Simplicius in the "De Caelo" of Aristotle.

> They move in the void and catching each other up jostle together; and some recoil in any direction that may chance, and others become entangled with one another in various degrees according to the symmetry of their shapes...and they remain together, and thus the coming into being of composite things is effected (*1*, p. 86).

These ideas of Leucippus were expanded by his pupil Democritus (*1*, pp. 128, 345). The lead given by the latter was later followed by Epicurus (*1*, p. 341), who described compound bodies in terms of "interlocking" atoms (solids) or of atoms encaged, as it were, by "shells" of interlaced atoms (liquids).

Unfortunately, only fragments of the writings of Epicurus on these topics survive. It is in Lucretius' poem, "De Rerum Natura," that the Epicurean notions are effectively preserved, though often modified or embellished by Lucretius himself. The following extracts from the poem in translation (*2*) illustrate how picturesquely Lucretius dealt with the subject of the binding of ultimate particles.

> And we see wine flow through the strainer as swiftly as you will; but, on the other hand, the sluggish olive-oil hangs back, because we may be sure, it is composed of particles either larger or more hooked and entangled one with the other, and so it comes about that the first-beginnings cannot so quickly be drawn apart, each single one from the rest, and so ooze through the single holes of each thing.
>
> Or, again, things which seem to us hard and compact, these, it must needs be, are made of particles more hooked one to another, and are held together close-fastened at their roots, as it were by branching particles. First of all in this class diamond stones stand in the forefront of the fight, well used to despise all blows, and stubborn flints and the strength of hard iron, and brass sockets, which scream aloud as they struggle against the bolts. Those things indeed must be made of particles more round and smooth, which are liquid with a fluid body: for indeed a handful of poppy-seed moves easily just as a draught of water; for the several round particles are not checked one by the other, and when struck, it will roll downhill just like water. Lastly, all things which you perceive flying asunder, like smoke, clouds and flames, it must needs be that even if they are not made entirely of smooth and round particles, yet they are not hampered by particles closely linked...

Not unnaturally, the problem of which the ancients were chiefly conscious was the way in which molecules cling together in the "condensed" liquid or solid phases. In addition to dealing with primary valence, modern theory is concerned with such effects, but they often involve attractive forces which are "residual" and weaker, by a factor of ten or more, than the primary bonding forces between atoms.

Despite the endeavor of Lucretius, interest in the problem of the binding of ultimate particles did not become widespread until the seventeenth century. This was mainly the result of the prolonged influence of the Aristotelian School, which was ever hostile to atomistic doctrines. In the early Christian age chemistry was almost exclusively an Arabian art based on the teaching of Aristotle. *Atomism* was generally rejected, therefore, and in such a system devoid of particles the question of their association clearly could not arise.

It was not until the early fifteenth century that atomism began to creep back into philosophy when Poggio rediscovered Lucretius, but for a long while it made little impression. In fact, the seventeenth century had arrived before any serious return to atomism began. The philosophy of Gassendi, especially, and the chemistry of Sala, Sennert, Van Helmont, and Galileo heralded the return. Descartes (*3*) and Locke (*4*) continued the process in terms of the corpuscle, at first deformable and even divisible. By the end of the seventeenth century the concept of the indivisible atom was fairly generally re-established.

With the return of atomism there was naturally a revival of interest in the binding agency between atoms. To begin with, the "hooked tails" of the atoms of

Epicurus and Lucretius reappeared. John Mayow (*5*), for instance, in 1674 postulated "branchy" particles that hooked on, one to another, and Robert Boyle considered the possibility of entangling particles.

He wrote (*6*),

> If we assign to corpuscles, whereof each element consists, a peculiar size and shape, it may easily enough be manifested that such differingly figured corpuscles may be mingled in such various proportions, and may be connected in so many several ways that an almost incredible number of variously qualified concretes may be composed of them.

The publication in 1687 of Sir Isaac Newton's "Philosophiae Naturalis Principia Mathematica" popularized gravitational forces, and these were quickly invoked to explain corpuscular attraction. It soon became clear, however, that the attractive forces between small particles could not be simply deduced from those between large masses, since small particles appeared to exhibit preferential attractions. Thus it was found that an iron strip, dipped in a solution of blue vitriol, received a deposit of copper. Blue vitriol was considered to be a compound of copper and sulfuric acid, and on the basis of these experiments Newton suggested that the attraction of iron for sulfuric acid was greater than that of copper. The idea was that copper deposited on the strip because iron had expelled it from the blue vitriol, and taken its place. Newton thus appreciated that his mechanics was unsuited to a description of the attractions between minute particles, and he appears to have been the first to grasp the idea of chemical affinity. Several other cases of such preferential affinities had been described, and they became known as *elective attractions* to the chemists of the time. The term sustains the notion of attractive forces of some kind in the atomic mechanism, but forces which are clearly not gravitational. This idea of cohesion, or chemical combination, through attractions between particles flourished early in the eighteenth century. It was less naïve than hypotheses about the shape of atoms, hooked or branched corpuscles, or "conspiring motions." Atomic shapes were not, however, immediately discarded. In the latter half of the eighteenth century they played an important part in Buffon's theory (*7*).

Buffon argued that if the moon and the earth were cylinders instead of spheres, "the various distances between their different small parts would be affected, and hence the total gravitational pull between the two bodies would alter." Admittedly the alteration would be small in this instance, since the distance between the centers of the earth and the moon is very large. On the other hand, if two bodies were very close, then differences in their shapes, by effectively varying the distribution of the attractive forces between them, would affect their total gravitational pull upon one another. Since the participating particles in chemical reactions are very close to one another, Buffon concluded that their shapes would greatly influence the total attractions between them. Thus the varying attractions between particles manifested in "elective attractions" might be explained. Buffon's theory was widely accepted. Indeed, men like Berthollet still adhered to it early in the nineteenth century.

Combining weights also began to claim increasing attention, for chemists saw in these a measure of chemical affinity. If a unit mass of a substance A combined with a large mass of B and a smaller mass of C it was presumed to have a greater affinity for B than for C. Ideas about the actual mechanism of combination also became less vague. In 1776 Bryan Higgins (*8*) individualized the atoms of elements by assigning to each an atmosphere of *caloric*. This notion was elaborated in 1789 by his nephew, William Higgins (*9*)[1] and later by Dalton in his "A New System of Chemical Philosophy," 1808–10. The atmospheres around the atoms were considered to form "buffers" against chemical union. Thus, oxygen and hydrogen did not unite by mere mixture because the affinities between the atoms were obstructed by the sheaths of caloric. When, however, an electric spark passed through the mixed gases, the caloric atmospheres were disrupted and *single* atoms of oxygen united with *single* atoms of hydrogen to form "compound atoms" of water or steam. Some caloric was lost in the process, but the residue surrounded each compound atom with an atmosphere that again protected the paired atoms against others. Thus the caloric atmospheres dispelled the vague idea of flocks of combining atoms, and fostered the notion of pairing between single atoms of each element. They were also a primitive expression of the idea of activation energies. Furthermore, since, according to Dalton, each atom of oxygen and hydrogen respectively in each compound atom had precisely the same weight, all samples of water contained the same elements in the same proportions by weight. Berzelius in 1831 (*10*) was the first to state explicitly this rationalization of the law of constant composition. This law had been tacitly assumed earlier by chemists like Rey, Black, and Lavoisier, and more positively expressed by Proust. As Berzelius recognized, it justified all attempts to determine chemical composition by analysis. It would obviously have been useless, for example, to analyze one sample of water if another sample might contain different elements, or even the same elements in different proportions. In the same way, the constant compositions of compounds other than water were related to the Higgins-Dalton mechanism of chemical combination.

NUMERICAL ASPECTS OF CHEMICAL BONDING

An even more convincing achievement of the new atomic theory appeared in its illumination of the law of multiple proportions. It was now easy to explain why, if two elements united in different propor-

[1] William Higgins also appears to have been among the first, if not actually the first, to devise graphic formulas of a kind. Credit for this is often given to Kekulé, whose symbolism did not appear until almost seventy years later. Furthermore, the symbolism of Higgins was comparatively advanced. It was a full-line bond symbolism on which was superimposed a quantitative notion of the varying strengths of different bonds.

tions to form several different compounds, the different weights of one element combining with a fixed weight of the other should bear a simple numerical relationship to each other. This merely meant that different *numbers* of atoms of one element were combining with the same number of the other element to give the different compounds. Since the atoms of each element all had the same weight the simple multiple ratios followed immediately.

Dalton thus diverted attention from the measurement of forces of affinity to that of the determination of combining ratios. By degrees each element had assigned to it a number representing its combining proportion in compounds. This number, designated the "proportion" by Davy, eventually became known as the *equivalent* of the element (*11*). Sir Humphry Davy (*12*), for example, assigned the following equivalents to various elements: hydrogen = 1, oxygen = 7.5, potassium = 40.5, chlorine = 32.9. Berzelius was also closely associated with the development of the theory of equivalents, which became firmly established as more and more compounds were examined.

But as more facts became available certain perplexing aspects of equivalents began to appear. Thus an equivalent seemed sometimes to be synonymous with an "atomic weight" and at other times merely a multiple of it. In water, for example, where eight parts by weight of oxygen combined with one part of hydrogen, the hydrogen equivalent weight was synonymous with its unit atomic weight, whereas the oxygen exhibited an equivalent which was only half its atomic weight of sixteen. There were other examples of the same sort of behavior. Confusion was rendered more acute by the inaccurate knowledge of atomic weights available at the time. It was only with the advent of the numerical concept of *valence*[2] and the introduction of a correct system of atomic and molecular weights by Cannizzaro in 1852 (*13*) that a final clarification was achieved.

Credit for the idea of a definite atomic attractive force, now known as valence, is due in the first place to Frankland. His classical paper, entitled "A new series of organic compounds containing metals," appeared in the *Philosophical Transactions* in 1852 (*14*). In it Frankland showed that individual atoms possess a definite power of entering into chemical combination. His own words may be quoted:

> ...no matter what the character of the uniting atoms may be, the combining power of the attracting element, if I may be allowed the term, is always satisfied by the same number of these atoms.

These ideas of Frankland were only slowly accepted but eventually by 1858 they were employed and extended by Couper (*15*) and by Kekulé (*16*).

The water molecule was again important in the development of the theory. Oxygen became known as a diad, or divalent, because *one* of its own atoms united with *two* atoms of hydrogen to form water. Chlorine, on the other hand, was termed a monad, or monovalent, because *one* of its own atoms united with *one* of hydrogen. Analogously nitrogen became trivalent or pentavalent because one of its own atoms united with three hydrogen atoms in ammonia or with the equivalent of five hydrogen atoms, as in ammonium chloride, for example. An element was x-valent if one of its atoms combined with x atoms of hydrogen, which was itself taken as the monovalent standard of reference. One atom of an x-valent element also united with one of another x-valent element if x were the same in both instances.

There were rival terms to that of *valence*, some of which are interesting in the light they shed on the mental associations of the time. To many, the so-called valences presented some analogy with saturation or neutralization. For instance, ten ounces of caustic potash required nearly nine ounces of sulfuric acid to neutralize it, while ten ounces of caustic soda required more than twelve. The alkali was saturated in either case by the particular quantity of acid and would accept no more. One atom of chlorine appeared to be rather analogously saturated by one atom only of hydrogen, but an oxygen atom required two atoms of hydrogen to saturate it. As late as 1875 the term *saturation* was being used as an alternative to that of *valence*. The synonym *atomicity*, which occasionally still occurs, was also widely used because of the influence of Frankland. Kekulé pressed for the recognition of the terms *monobasic*, *dibasic*, and so on, to signify the combining power of the various atoms.

The really important thing, however, about the concept of valence or atomicity was that it clarified the relationship between the atomic weight of an element and its equivalent; it was clear that valence could be defined simply as the ratio of the former to the latter.

THE MECHANISM OF CHEMICAL BONDING AND ITS ORIGINS

In the developments described above, which replaced to some extent interest in the older qualitative questions of why and how particles combined, valence was merely a number. However, concurrently with the development of this numerical concept, there also arose more precise ideas about the actual mechanism of combination.

In 1790 Volta discovered a chemical method of producing an electric current, and in 1800 he published a description of the voltaic pile (*17*). In 1801 Nicholson and Carlisle (*18*) discovered that water could be electrolytically decomposed, hydrogen being evolved at the negative, and oxygen at the positive pole. In 1803 Berzelius and Hisinger (*19*) carried out similar experiments on aqueous salt solutions. These studies led immediately to attempts by Davy and Berzelius to relate the electrical dualism or polarity of atoms to chemical affinity.

[2] The term "*valence*," denoting atomic combining capacity, did not finally appear until 1868. In 1865 Hoffman, in his "Introduction to Modern Chemistry," proposed the term "quantivalence." Three years later it was shortened to "valence" by Wichelhaus (*Ann. Supp.*, 6, 257 (1868)).

Davy was the first to expound an electrochemical theory, in 1807 (*20*). He pointed out that "hydrogene, the alkaline substances, the metals, and certain metallic oxides are attracted by negatively electrified metallic surfaces," whereas "oxygene and acid substances are attracted by positively electrified metallic surfaces." These observations were correlated with the results of electrification by friction. For instance, when sulfur was rubbed against copper, the sulfur became negatively and the copper positively charged, exhibiting the same states of electrification as in electrolytic decomposition. Davy inferred that chemical combination was due to the electrical attraction of oppositely charged bodies, and that chemical affinity was proportional to the difference in the electrical condition of the bodies.

Berzelius published the main outlines of his electrochemical theory in 1812 and a detailed exposition appeared in 1819 (*21*). His views were largely in agreement with those of Davy. An important difference was that Davy considered that charges were developed *only when two suitable substances approached one another*, while Berzelius maintained that the charges were already present in the atoms, each atom possessing both positive and negative electricity, more or less concentrated at poles in the atom. Berzelius also suggested that each atom had a predominant state of electrification depending on the relative amounts of positive and negative electricity possessed by it. He arranged the elements in what corresponds to an electrochemical series in which oxygen was the most negative and the alkali metals the most positive. Finally, he considered that after atoms had combined, the electrical charges in simple compound atoms were not necessarily neutralized. The resultant "compound atoms" also had electrical properties, and could in many cases combine again to form still larger compound atoms. For example, sodium oxide was considered to be a compound of the first order possessing free *positive* electricity, while SO_3, also of the first order, possessed free *negative* electricity. These two compounds in turn combined to form the second-order compound sodium sulfate, in which the positive and negative electricity might still remain unneutralized. Thus sodium sulfate could combine with other similar substances of opposite polarity to form compounds of the third order, the double salts. These embryonic ideas anticipated Werner's theory of complex formation and the later electronic interpretation based on the presence of unshared electrons.

CHEMICAL BONDING IN ORGANIC COMPOUNDS

In a paper on substitution read in 1834 Dumas (*22*) directed attention to the fact that, in the chlorination of methyl alcohol, hydrogen was replaced by chlorine without any fundamental change in the chemical properties of the molecule. The phenomenon of substitution was soon discovered to be quite general. Dumas, Laurent, and others showed that hydrogen was replaceable, not only by chlorine, but also by bromine, iodine, oxygen, and even carbon. Dumas finally made the general assertion that "every chemical compound forms a complete whole. Its chemical nature depends primarily on the arrangement and number (type groupings) of the constituent atoms, and to a lesser degree on their chemical nature."

This assertion that a negative chlorine atom could take the place of a positive hydrogen atom in an organic radical was in sharp contradiction to the electrochemical dualism of Berzelius. The ensuing controversy between the Swedish and French schools was protracted, and at times acrimonious, and the story of how the debate swayed to and fro as the great scientific personalities of other countries took sides and marshaled facts is by no means dull. So much bitterness resulted that each of the contending parties ended by completely ignoring the views of the other.

As is not infrequently the case in bitter disputes, both sides were partly wrong and partly right. In fact, the controversy marked a parting of the ways for inorganic and organic chemistry. Inorganic chemists continued to find the electrochemical theory fruitful and to develop it. Organic chemists developed the arithmetical concept of valence and type structural chemistry, and completely neglected any discussion of the nature of chemical affinity. This divergence of approach persisted for a considerable time and Maxwell probably expressed the opinion of many when he wrote (*23*) "the fact that every chemical compound is not an electrolyte shows that chemical combination is a process of a higher order of complexity than any purely electrical phenomenon."

THE DEVELOPMENT OF BOND SYMBOLISM

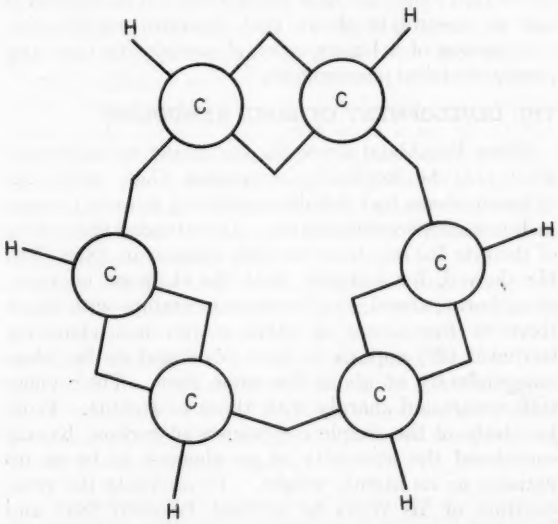
When Frankland wrote his first paper on valence in 1852 (*14*) he implicitly recognized that, while the different atoms had definite combining powers, in some instances these were variable. He extended this notion of definite *but sometimes variable* valence in 1858 (*24*). He showed, for instance, that the elements nitrogen, phosphorus, arsenic, and antimony combine with either three or five atoms of other monovalent elements. Gerhardt (*25*) appears to have developed similar ideas independently at about the same time. Their views thus contrasted sharply with those of Kekulé. From his study of the simple compounds of carbon, Kekulé considered the atomicity of an element to be as invariable as its atomic weight. To facilitate the presentation of his views he devised, between 1851 and 1861 (*26*), a kind of graphic symbolism in which atoms of different valence were represented as of different size. Thus hydrogen chloride, water, ammonia, methane, and hydrogen cyanide were represented by the following graphic formulas:

These symbols were an important advance at the time,

since they attempted to picture the exact relations of the atoms in the molecules. In conjunction with Kekulé's notion of the ability of carbon atoms to combine with each other, they provided the transition from the theory of types to that of *structural chemistry*. Couper (*27*) about the same time used similar formulas in which the atom symbols were joined by means of hyphens. This method was extended by Crum Brown (*28*). Soon after, the initial letters B, C, N, O, H, etc., originally used by W. Higgins and later by Berzelius, were readopted by Erlenmeyer to signify the different elements. In 1866 Frankland (*29*) finalized the method of formulation now in use and introduced the term "bond."

DOUBLE AND TRIPLE BONDS

While Kekulé was able to demonstrate the constant valence of carbon in saturated compounds, the matter was not quite so simple for unsaturated molecules. Several attempts were made by him to elucidate the nature of unsaturation, before his brilliant conception of the ring structure of benzene substantially clarified the problem. To maintain the tetravalence of carbon in such a ring the notion was advanced that the carbon atoms were held together by the mutual exercise of three valence units each. The following graphic formula, published in 1865 (*30*) pictures for the first time double bonds and a ring of six carbon atoms.



This notion of the *double bond* was fundamental to the future development of the structural chemistry of unsaturated compounds. Kekulé's views about the constancy of valence in general were too narrow, but the thoroughness and ingenuity with which they were maintained resulted in vital contributions to the structural chemistry of carbon compounds.

STEREOCHEMICAL ASPECTS OF CHEMICAL BONDING

Structural formulas led to important developments in the interpretation of the many instances of isomerism[3] then known. Obviously one basis of interpretation of isomerism was the alternative possible arrangements of atoms in isomeric molecules. It was soon inferred that ammonium cyanate and urea had different molecular arrangements of the same atoms. Observed differences in properties of certain isomeric substances could not, however, be explained in this way. One group of substances such as tartaric acid, aspartic acid, and malic acid was known in different forms which we now term optical isomers. In 1863 lactic acid was added to the list by Wislicenus (*31*) who showed that the two optical isomers were *both* α-hydroxypropionic acid. Wislicenus stated then, and expounded his views more fully later (*32*), that such isomers could "only be explained by supposing that the difference in the isomeric molecules is to be accounted for by a difference *in the spatial arrangement of their atoms.*"

While Wislicenus was developing these concepts another apparently different problem of isomerism was recognized. For a compound like CH_2Cl_2 it appeared that there should be *two* arrangements of the atoms: one with the like and the other with the unlike atoms adjacent. No such isomers, however, appeared to exist.

In 1874 van't Hoff (*33*) and Le Bel (*34*) resolved both the problem of the missing isomers and of optical isomerism by considering that atoms in a molecule occupied *space*. Chemical bonds need not necessarily be in a plane. A further step, made by van't Hoff, was to represent the four valences of carbon as directed toward the corners of a regular tetrahedron.[4] The views which van't Hoff developed over a number of years were set out in detail in 1878 (*35*). The phenomenon of optical isomerism was interpreted as due to different arrangements of the atoms *in space* around a central carbon atom. The nonexistence of isomers for molecules like CH_2Cl_2 follows directly from the regular tetrahedral model assumed. Following this success the concept of *directed valence* and the science of stereochemistry soon became widely established.

RESIDUAL VALENCES

Kekulé's notion of the carbon-carbon double bond led to the elucidation of the structures of unsaturated compounds in terms of multiple bonds in general, and the concept of the *triple bond* soon followed.[5] Meanwhile a different type of unsaturation was that involved in the formation of molecular compounds such

[3] The term *isomerism* was first used by Berzelius in 1832 (*Jahresber.*, **11**, 44).

[4] Actually Kekulé had suggested in 1867 (*Z. Chem.*, N.S., **3**, 217 (1867)) that the four units of affinity of a carbon atom projected from the surface in the direction of the four faces of a tetrahedron, but he does not appear to have followed up the idea.

[5] The idea of double and triple bonds did not readily receive general acceptance. As late as 1904 Hinrichsen (*Z. physik. Chem.*, **39**, 305 (1902); *Ann.*, **336**, 175 (1904)) preferred to write ethylene and acetylene as single-bonded systems of trivalent and divalent carbon, respectively. He argued, for instance, that whereas according to Baeyer's theory of strain acetylene should be very unstable, in fact it is not.

as hydrates and complex salts. It thus appeared that certain molecules, although saturated numerically in the Kekulé sense, still possessed some *residual affinity* which enabled them to combine with other equally saturated molecules. The nature of this residual affinity presented an intriguing problem. Some conflict was apparent between the notion of it and measurements of the quantity of electricity involved in chemical combination. The observation that different compounds, such as sodium chloride and silver chloride, showed different residual affinities was generally taken to imply that the quantity of electricity collected at the point of union between two atoms increased with the strength of their affinity. Such an idea, as Helmholtz clearly stated in his Faraday lecture of 1881 (*36*), ignored the significance of Faraday's work on electrolytic decomposition. Helmholtz said:

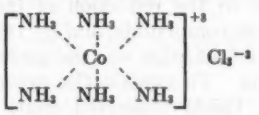
> Faraday demonstrated that so far as electricity came forth in electrolytic decomposition *its quantity did not at all depend on the degree of affinity*.... The most startling result of Faraday's law is perhaps this. If we accept the hypothesis that the elementary substances are composed of atoms, we cannot avoid concluding that electricity also, positive as well as negative, is divided into definite elementary portions which behave like atoms of electricity.

Helmholtz drew the conclusion that chemical union is somehow associated with these positive and negative "atoms" of electricity. In the same year G. J. Stoney (*37*) published a lecture originally read before a meeting of the British Association at Belfast in 1874. In the course of this lecture he stated: "For each chemical bond which is ruptured within an electrolyte a certain quantity of electricity which is the same in all cases traverses the electrolyte." Stoney estimated the value of this elementary electrical charge to be 0.3×10^{-10} absolute electrostatic unit. The electron and electronic interpretations of valence were beginning to appear on the horizon. The latter remained comparatively undeveloped, however, for some considerable time. For instance, Werner, in developing his theory of chemical affinity in 1891, assumed that when one atom is subjected to the affinity demands of another, varying amounts of this affinity are harnessed to produce a valence bond. Hence also, varying amounts of residual affinity are left free. Werner believed the property of attraction to emanate from the center of each atom and to be evenly distributed over its surface (*Bindeflache*). He considered the Kekulé-van't Hoff conception of four individual valences operating in space as untenable. This opinion was maintained for over twenty years, and is expressed strongly in his "New Ideas" published in 1911.[6]

Werner's views were descriptive rather than quantitative. He considered that the affinity of a central atom might be partially saturated by direct union with other atoms (*primary valence*, symbolized by unbroken lines), to produce ordinary molecules. Often there remained a residual affinity which caused further chemical union (*auxiliary valence*). These auxiliary valences, symbolized by dotted lines, had their own peculiar restricting conditions. They might bind the central atom to neutral molecules like water and ammonia, or to ions. The latter type of union required the presence of another ion. For instance, in the attachment of a hydrogen ion to ammonia, an anion, such as the chloride anion, was also involved.

According to Werner, the atoms or groups attached directly to the central atom were very firmly bound. He described them as being in the "coordination group" or "first sphere." They were not "ionised." The other atoms of the molecule were described as being in the "second sphere." These might be ionized. Werner enclosed the coordination group in square brackets and termed the maximum number of atoms or groups which might be directly bound to the central atom either by primary or auxiliary valence the *coordination number*. The magnitude of this number was thought to be governed largely by the amount of space constituting the first zone. It was frequently either four or six.

Werner's theory may be illustrated by the cobalt chloride ammines. The substance $CoCl_3 \cdot 6NH_3$ gives three chloride ions in aqueous solution. These may be precipitated with silver nitrate. On the other hand, the ammonia molecules are not removed when the solution is treated with sulfuric acid. Werner described this behavior by the formula:



Series of such complexes are known. Significant examples include:

(1)	$[Co(NH_3)_6]Cl_3$	(4)	$[Co(NH_3)_3(NO_2)_3]$
(2)	$[Co(NH_3)_5Cl]Cl_2$	(5)	$[Co(NH_3)_2(NO_2)_4]K$
(3)	$[Co(NH_3)_4Cl_2]Cl$	(6)	$[Co(NO_2)_6]K_3$

In each compound the cobalt exhibits a coordination number of six. Compound (4) is a nonelectrolyte since the second sphere is empty. In the first three compounds the coordination complex is a cation, while in the last two it is an anion.

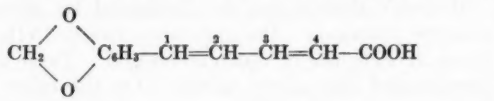
It was only when Werner was able to predict the occurrence of optical activity in certain of these metal complexes, and to verify his predictions by experiment in 1911, that his views obtained fairly general support. They also applied to problems of structural organic chemistry without bringing much that was new.

RESIDUAL VALENCE OF UNSATURATED COMPOUNDS

A quite different attempt to refine numerical concepts of valence by introducing the notion of residual valence

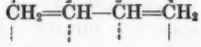
[6] According to Waters and Lowry ("Physical Aspects of Organic Chemistry," 1936, p. 7) Werner appears to have adopted the views of Lossen (1880), who states that "the valency of an atom is merely a number which expresses how many atoms are present in its combining zone" and of Claus (1881), who said that "the hypothesis that valency in multivalent atoms is a pre-existing force acting in definite units is as unfounded as it is unnatural."

was put forward by Thiele in 1899 (*38*). Kekulé had originally assumed that "free" valences were present in unsaturated compounds. But these "free" valences were always observed to occur in pairs; all attempts to prepare methylene resulted in the formation of ethylene. Hence there arose the assumption that the so-called free valences of unsaturated compounds mutually saturated each other. As already stated above, this gave rise to the symbolic notion of double and triple bonds between atoms. However, the observed behavior of unsaturated compounds was not completely represented by ordinary multiple bond symbolism. For example, in the series $CH_3—CH_3$, $CH_2=CH_2$, $CH\equiv CH$, a firmer binding is indicated by the symbols for ethylene and acetylene than for ethane. While this firmer binding is supported by thermochemical studies of bonds it does not take account of the greater reactivity of unsaturated compounds. The earliest attempt to resolve the paradox of stronger bonding combined with greater reactivity was the work of von Baeyer (*39*), who presented his strain theory in 1885. But while this provided a plausible interpretation of the enhanced reactivity of substances possessing one double or triple bond, Thiele pointed out that a similar interpretation of the properties of substances possessing conjugated double bonds was inadequate. Fittig (*40*) had shown in 1888 that in the reduction of piperic acid,

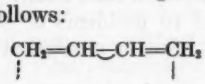


hydrogen adds primarily in the 1,4 and not in the 1,2 or 3,4 positions. Parallel observations had been made by von Baeyer in the reduction of terephthalic acid and several other compounds, and by 1899 the evidence indicated that 1,4 addition was the general rule for conjugated systems. To explain the prevalence of these 1,4 additions, Thiele suggested that, in a multiple linkage, only the first of the valence bonds uses up as much atomic affinity as an ordinary single bond. Thus a large but variable amount of affinity is left free. This residual free affinity Thiele termed the *partial valence*.

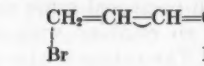
Consider, for example, the addition of bromine to butadiene,



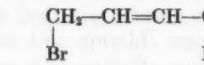
where the dotted lines represent the partial valences of Thiele. Thiele argued that, as is the general rule in the formation of true double bonds, the partial valences on the singly bound carbon atoms 2 and 3 mutually saturate each other across the single bond. The result was pictured as follows:



The terminal carbon atoms are thus left with their excess of affinity or partial valences, and the phenomenon of 1,4 addition follows naturally. The incoming atoms were considered by Thiele first to attach themselves loosely to the two carbon atoms:



There then follows a redistribution of affinity with the formation of the final product:



In the working out of his ideas, Thiele displayed an intuitive anticipation, real if rather vague, of the concept of resonance.

ELECTRONIC THEORIES OF VALENCE

While theories of variable and residual affinity held the field, the dualistic electrostatic theory of Berzelius was not entirely ousted. As has been discussed, Helmholtz was convinced of the electrical nature of affinity by 1881 (*36*), but it was mainly the Arrhenius theory of electrolytic dissociation that gave impetus to a renewed interest in the views of Berzelius. It became apparent that atomic linkages are of two kinds, one ionizable and the other not, one presumed due to electrostatic attraction, and the other unexplained. A possible third type of link, Werner's coordinate link, seemed to be capable of uniting apparently saturated molecules.

At the beginning of the present century the electron was recognized as a constituent of all atoms, and, following the lead of J. J. Thompson, this knowledge was quickly applied to the problem of chemical binding. In 1902 Lodge (*41*) made the important simplifying suggestion that only *negative* electrons were transferred in chemical combinations, negative charges on atoms representing the acquisition of electrons and positive charges the loss of electrons. Previous to this the participation of both positive and negative "atoms" of electricity had been generally invoked. Lodge, further, was the first to propose that electrons in one atom might come within the sphere of influence of another atom in combination with it. Such an idea may surely be regarded as the germ of subsequent theories of electron-sharing in nonionized compounds.

Simultaneously with the emergence of such fundamental notions Abbegg and Bodlander (*42*) were drawing attention to interesting valence trends within the framework of the periodic system.[7]

These authors in 1899 replaced the concept of affinity between atoms by that of affinity of atoms for electrons and in 1904 Abbegg (*43*) published his "rule of eight."

[7] Of course, the importance of the periodic classification to valence theory was recognized from the beginning. Mendeleev himself observed that the elements of any particular group exhibited a valence characteristic of the group as a whole. It thus became evident that if an element were placed correctly in the periodic table (*i. e.*, if its atomic weight were known accurately) the group into which it fell provided a valuable clue to its valence. The case of beryllium is a well known historical example of the value of the periodic table in this respect.

Group	*1*	*2*	*3*	*4*	*5*	*6*	*7*
Normal valence (v)	+1	+2	+3	+4	−3	−2	−1
Contravalence (v')	−7	−6	−5	...	+5	+6	+7

"Every element can have a *normal valency* and also a valency of opposite sign, called a *contravalency.*" The arithmetical sum of the maximum values of these two valences for any element is always eight. Abbegg illustrated this rule by the table based on Mendeleev's relation between the valence of an element and its position in the periodic table.

Also in 1904, Drude (*44*) made the following comment on Abbegg's work: "Abbegg's positive valency number v, whether it is a normal or a contravalency, signifies the number of loosely attached negative electrons in the atom: his negative valency number v' means that the atom has the power of removing v' negative electrons from other atoms, or at least, of attaching them more firmly to itself." Sidgwick (*45*) has remarked, "considering the time when this was written, it shows a very remarkable advance on current theories."

A definite relationship between valence and the periodic system needed, however, the formulation of the Rutherford nuclear atom, the Bohr electronic orbits, and Moseley's elucidation of the role of *atomic number* and it was not until 1916 that a significant interpretation of the electronic mechanism of valence was put forward independently by Kossel (*46*) and by Lewis (*47*). It is then that the modern outlook may be said to have really begun.

ACKNOWLEDGMENT

I wish to thank Professors T. S. Wheeler and A. R. Ubbelohde for their interest and discussion.

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(32) Wislicenus, J., *ibid.*, 167, 343 (1873).
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(35) van't Hoff, J. H., "Ansichten über organische Chemie," 1878.
(36) Helmholtz, H. von, *J. Chem. Soc.*, 39, 285 (1881).
(37) Stoney, G. J., *Phil. Mag.*, 11, 384 (1881).
(38) Thiele, *Ann.*, 306, 87, 369 (1899).
(39) von Baeyer, A., *Ber.*, 18, 2278 (1885).
(40) Fittig, R., *Ann.*, 227, 46 (1885).
(41) Lodge, O., *J. Inst. Elec. Engrs. London*, 32, 45 (1902–3); "Modern Views on Matter," Romanes Lecture, Oxford, 1903; "Electrons," 1906.
(42) Abbegg and Bodlander, *Z. anorg. Chem.*, 20, 453 (1899).
(43) Abbegg, *ibid.*, 39, 330 (1904).
(44) Drude, *Ann. Physik*, 14, 722 (1904).
(45) Sidgwick, N. V., "The Electronic Theory of Valency," Oxford Univ. Press, London, 1927, p. 53.
(46) Kossel, *Ann. Physik*, (4), 49, 229 (1916).
(47) Lewis, G. N., *J. Am. Chem. Soc.*, 38, 7762 (1916).

CORRECTION

In "The mechanism of diazotization," (this Journal, 31 344 (1954)) an arithmetical error in the pK's of *p*-aminobenzylamine has led to a consequent error in the relative amounts of free base, mono-, and di-salts presented in the table. The values of pK should be 5.30 and 9.38. Of 10^{12} molecules of aminobenzylamine, there would be present as free base at pH 1, 2 molecules; at pH 2, 208; at pH 3, 2.07×10^4; at pH 4, 1.98×10^6. As mono-salt at the same pH's there would be 4.99×10^8; 4.00×10^9; 4.97×10^{10}; and 4.76×10^{11} molecules respectively.

Revision of these values does not alter the argument, or the conclusion, that it is the free base which takes part in diazotization, and not the salt.

—*Richard G. Gillis*

MOLECULAR ASSOCIATION OF ORGANIC SUBSTANCES[1]

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MOLECULAR compound formation between neutral substances has long intrigued organic chemists, but for the past decade we have been on the crest of a wave of interest in such substances, as well as in molecular association in solution. The solid compounds have become of considerable value, being useful for the isolation, purification, and molecular weight determination of amines and hydrocarbons, while the complexes in solution are leading to a better understanding of the nature of valence electrons in certain types of molecules. Solid molecular addition compounds, such as the picrates and quinhydrones, and association in solution of hydroxy and amino compounds through hydrogen bonds have been known for many years, but the forces of attraction between the component molecules in these and other molecular addition complexes are still not completely understood. Molecular association has been found to be so prevalent and is of such wide current interest that it offers a timely topic for discussion here.

CLATHRATES

The most recent and perhaps most fascinating of the solid complexes arising through molecular association are the *clathrates*.[2,3] These are crystalline compounds which arise by crystallization of certain substances in a regular, open-lattice, cagelike structure so that other molecules are entrapped in the open cavities. For example, upon crystallization, hydroquinone or catechol can enclose small molecules like sulfur dioxide, formic acid, methanol, nitric oxide, or oxygen. The imprisoned molecules can be liberated by any process that destroys the crystal framework, such as melting or the addition of organic solvents to dissolve the quinol molecules. There is no apparent chemical attraction in the usual sense between the components of these complexes. For example, the inert rare gases, argon, xenon, etc., can be stored in the form of their quinol complexes from which they may be regenerated by dissolution of the quinol. Thus the complexes offer a convenient means of dispensing the rare gases for student laboratory use. Also, it has been found that some of the physical properties of the entrapped gas, such as its paramagnetism, are not changed appreciably, and hence the complexes provide a method of studying the properties of, for instance, nitric oxide at low temperatures without its liquefaction or dimerization as otherwise would occur at 121°K.

Another group of clathrate compounds is the urea complexes.[2,4] Urea forms complexes with almost any type of straight-chain organic molecule, *e. g.*, hydrocarbons, alcohols, esters, etc., and the complexes are decomposed by melting, dissolving out the enclosed molecules with organic solvent, or by dissolving away the urea with water. They are easily prepared, a common method being to dissolve separately urea and some organic compound in hot methanol, mix the two solutions, and allow the crystals to settle out upon cooling. The urea molecules crystallize in the form of helices by head-to-tail hydrogen bonding and entrap organic molecules down the center channels. The diameter of the tubular space is fixed and only molecules with the proper dimensions to fit in the channels will form complexes. It so happens that hydrocarbon straight chains fit snugly but branched chains are too large. For each type of functional group there is a certain minimum chain length for complex formation. For example, it is eight carbon atoms for esters, seven for alcohols, six for hydrocarbons, five for acids, etc. Owing to the dependence of this preferential complex formation upon molecular *shape* and *size*, various mixtures can be resolved. As illustration, it is well known that branched-chain hydrocarbons give better motor fuels than do straight-chain hydrocarbons, and removal of the latter through their urea complexes raises the octane rating of some petroleum products. One only has to mix the fuel with a saturated aqueous urea solution and, after allowing to stand for some time, to run off the upgraded fuel.

Many fats and waxes have little or no commercial value because of high fatty acid content. Removal of the free acid is seldom undertaken because of the formation of emulsions which are hard to handle. Treatment with moist urea, however, will easily remove the fatty acids by means of their complexes, leaving behind the now usable oil or wax, which consists of cyclic and branched-chain compounds. In other instances, fatty

[1] Based on talks given, spring, 1954, before the Lund Chemical Society, University of Lund, Lund, Sweden, and the Danish Chemical Society, University of Copenhagen, Copenhagen, Denmark.

[2] TRUTER, E. V., *Research (London)*, **6**, 320 (1953).

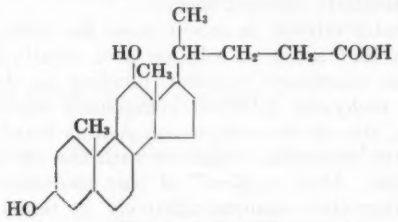
[3] SCHLENK, W., JR., *Fortschr. chem. Forsch.*, **2**, 92 (1951); J. W. SMITH, *Science Progr.*, **38**, 698 (1950); H. M. POWELL, *Endeavour*, **9**, 154 (1950).

[4] ZIMMERSHIED, W. J., R. A. DINERSTEIN, A. W. WEITKAMP, AND R. F. MARSCHNER, *Ind. Eng. Chem.*, **42**, 1300 (1950); O. REDLICH, C. M. GABLE, A. K. DUNLOP, AND R. W. MILLAR, *J. Am. Chem. Soc.*, **72**, 4153, 4161 (1950).

acids are isolated from crude oils in relatively pure condition by means of their urea complexes. Also, naturally occurring mixtures of stearic, oleic, and linoleic acids may be resolved by fractional complex formation. Unsaturation decreases complex stability so that treatment of this mixture of acids with a limited amount of urea solution first removes the stearic acid. Subsequent treatment with another limited portion of urea solution will then take out the oleic acid. In this fashion, such mixtures have been separated to yield the components in 90–95 per cent purity. Also, substances may be stored in the form of their urea complexes for protection. Thus, the auto-oxidation of oleic acid is completely stopped by storing the acid in the form of its complex.

Thiourea forms the same type of crystalline complexes as does urea, but the channel of the helix is larger. It is too large to hold straight-chain carbon compounds, excepting the very long chains, but will retain branched-chain and cyclic derivatives. Therefore, thiourea and urea can be used in a complementary fashion. It can be seen that these clathrate complexes offer a powerful tool for resolving mixtures; their importance in this regard is increasingly being recognized. For instance, the separation of benzene and cyclohexane, whose boiling points are less than one degree apart, is no elementary laboratory task. Now it can be done by simply treating the mixture with wet thiourea and filtering out the cyclohexane complex. The cyclohexane can be recovered from its complex by shaking the latter in water and separating the thereupon floating cyclohexane with a separatory funnel.

It has been known for some time that the steroid desoxycholic acid forms these clathrate complexes with



Desoxycholic acid

many classes of organic compounds, including hydrocarbons.[5] The complexes, called choleic acids, amazingly dissolve in alkali without decomposition and thus offer a means of getting hydrocarbons into aqueous solution.

Another significant use of clathrate compounds is for the resolution of racemic mixtures.[6] The complexes of a pair of optically active isomers have different solubilities, so that it is possible to carry out a resolution. In this way, *dl*-2-chlorooctane and *dl*-sec-butyl bromide, for example, have been resolved. It is noteworthy that resolution does not depend upon the presence of a chemical functional group in the racemate nor upon an optically active reagent for effecting the resolution.

It is probable that many other types of clathrate compounds will be found that will be capable of effecting specific separations. For example, ammonia solutions of nickel cyanide form complexes with benzene, pyridine, pyrrole, and phenol, but molecules with a slightly different shape, such as toluene and chlorobenzene, cannot be accommodated in the crystal cavities.[7] In this way, pyridine might be separable from picoline.

SOLID MOLECULAR ADDITION COMPOUNDS[8]

A second class of solid molecular association compounds is what has been known for years as the molecular addition compounds, such as the picrates and quinhydrones.[9] Unlike the clathrates, which crystallize in molar ratios ranging from 2 to 20 molecules of the crystal-lattice component to one of the enclosed molecules, the molecular addition compounds usually form in 1:1 molar ratios of the two components. One component, which we will designate as A, is always an electrophilic substance, *i. e.*, electron-seeking, and the other component, referred to as B, is an electron-donor compound. From a number of diffraction studies it has been found that the crystals of molecular addition compounds consist of alternate layers of the two components, about 3–3.5 Å. apart.[10] It is often embarrassing to organic chemistry teachers to try and give the structural formula of picrates or of quinhydrone to an elementary organic class, because the bonding in these compounds is not easily designated by symbols, as are covalent or simple ionic bonds. The most attractive view at this time on the bonding forces in these compounds is that there is a complex bond of partial ionic and partial covalent character between the two components.[11] This concept has been advanced in various terms by several persons, each one of whom held the notion that there is a loose electronic intermolecular interaction between components, which closely resembles an *intermolecular* coordinate covalent bond. Such a bond has covalent and ionic character; however, the covalent bonding is not between any two specific atoms but between the two molecules as a whole. In modern terminology, the situation can be described as a

[5] FIESER, L. F., "The Chemistry of Natural Products Related to Phenanthrene," 2nd ed., Reinhold Publishing Corp., New York, 1937, pp. 129, 368.

[6] POWELL, H. M., *Nature*, 170, 155 (1952); W. SCHLENK, JR., *Experientia*, 8, 337 (1952).

[7] RAYNER, J. H., AND H. M. POWELL, *J. Chem. Soc.*, 1952, 319.

[8] This discussion excludes the metal halide-ammonia derivative type salts, $AlX_3:NR_3$, which are formed essentially by acid-base neutralization in the Lewis sense.

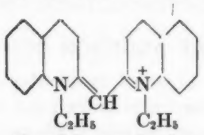
[9] FERGUSON, L. N., "Electron Structures of Organic Molecules," Prentice-Hall, Inc., New York, 1952, pp. 47–52; G. W. WHELAND, "Advanced Organic Chemistry," 2nd ed., John Wiley & Sons, Inc., New York, 1949, p. 69.

[10] WALLWORK, S. C., AND T. T. HARDING, *Nature*, 171, 40 (1953); W. S. RAPSON, D. H. SAUNDER, AND E. T. STEWART, *J. Chem. Soc.*, 1946, 1110; H. M. POWELL AND G. HUSE, *Nature*, 144, 77 (1939); *J. Chem. Soc.*, 1943, 153, 435.

[11] MULLIKEN, R. S., *J. Am. Chem. Soc.*, 64, 811 (1952).

resonance between the no-bond form A, B and the coordinate covalent structure $^-A—B^+$, where the covalent bonding involves an overlapping of the molecular pi orbitals in a perpendicular direction between the component molecules.

The strongest evidence for this viewpoint is provided by spectroscopic data.[12] With polarized light, it is found that absorption of light is greatest when the electric vibrations are perpendicular to the parallel planes of the two components.[13] The same thing is true concerning the light absorption by the polymer of pseudoisocyanine.[14] In aqueous solution, these flat ions will polymerize face to face to form long threads



Pseudoisocyanine ion

and it is found that they show the greatest light absorption in the direction along the threads. In a similar fashion, the molecular addition compounds have a large polarizability perpendicular to the planes of the components which leads to strong absorption in the near ultraviolet and visible regions of the spectrum.

It is noteworthy that steric requirements may reduce the stability of these molecular addition compounds.[15] If either molecule is nonplanar, the distance between planes is increased, which decreases both the overlap of molecular orbitals and polarization of B by A. Therefore, ionic and covalent bonding forces are much weaker. For example, *trans*-stilbene forms a picrate but the nonplanar *cis* isomer does not, and β-phenylnaphthalene forms a picrate but the alpha isomer does not. In the latter compound, the *peri* hydrogen atom prevents the phenyl group from being coplanar with the naphthalene ring.

The molecular addition compounds serve in a useful capacity in several ways. They have long been used for the isolation, purification, and identification of amines and polynuclear hydrocarbons,[16] particularly the picrates. The use of quinhydrone in electrolytic cells is quite familiar. Recently the picrates have been used also for determining molecular weights. It was discovered that many amine-picrates have a strong absorption band near 3800 Å. and of the same intensity.[17] Hence, by the use of the Bouguer-Beer law, $A_x = a/cd$, where A_x is 38,440, c is the picrate concentration, d is the cell thickness, and a is the observed solution absorbancy, the molecular weight may be determined. This has led to very good results with a minimum of inconvenience. Equivalent weights of picrates can also be determined by titration in aqueous[18] or nonaqueous[19] media with satisfactory results.

ASSOCIATION IN SOLUTION

In recent years, it has been observed that association between many combinations of substances occurs in solution to give dimers and polymers. Most common are the hydroxylic and amino compounds that associate through the familiar hydrogen bonds. This association is due to dipole-dipole attractive forces and has a marked effect upon solubilities, vapor pressures, and infrared spectra of substances. However, since hydrogen bonding has been reviewed frequently[20] it will not be discussed here. Nitro, carbonyl, and sulfonyl compounds associate too, owing to dipole-dipole forces, but to a smaller degree. This association has the same qualitative effect upon properties as does hydrogen bonding. With the exception of a few outstanding cases, as, for example carboxylic acids and amides, association from dipole-dipole attractions leads to polymers and causes little alteration of the ultraviolet or visible spectra of the monomers, although distinct changes occur in the infrared spectra. Accordingly, such association cannot be studied by ultraviolet spectroscopy[21] (do not confuse *inter*molecular H-bonding with *intra*molecular H-bonding; the latter produces marked spectroscopic effects in the ultraviolet and visible regions of the spectrum) but can be studied through measurements of immiscible solvent distribution, Trouton's constants, and particularly infrared spectra.

Of greater interest in recent years has been the association that arises in solution as a result of intermolecular coordinate covalent bonding as described for the molecular addition compounds above. For example, the electron-donor compounds listed in the table form molecular complexes with the electrophilic substances. Most studies[22] of this association have been interpreted semiquantitatively in terms of the formation of 1:1 molecular addition complexes in solution, and, in a few cases, of complexes of other molar ratios between the electron donor and electrophilic reagents. The complexes are characterized by exhibiting ultraviolet or visible absorption not shown by their components. For instance, iodine vapor and solutions of iodine in inert solvents like heptane or carbon tetrachloride are violet, while solutions of iodine in aromatic compounds are brown, so the brown color is

[12] McConnell, H., J. S. Ham, and J. R. Platt, *J. Chem. Phys.*, **21**, 66 (1953); H. Tsubomura, *Bull. Chem. Soc. Japan*, **26**, 304 (1953).

[13] Nakamoto, K., *J. Am. Chem. Soc.*, **74**, 1739 (1952).

[14] Jelley, E. E., *Nature*, **139**, 631 (1937); G. Scheibe, *Ber.*, **58**, 586 (1925).

[15] Orchin, M., *J. Org. Chem.*, **16**, 1165 (1951).

[16] Orchin, M., and E. O. Woolfolk, *J. Am. Chem. Soc.*, **68**, 1727 (1946); Staff of Hopkin and Williams Research Laboratory, "Organic Reagents for Organic Analysis," 2nd ed., Chemical Publishing Co., Brooklyn, **1950**, p. 13.

[17] Cunningham, K. G., W. Dawson, and F. S. Spring, *J. Chem. Soc.*, **1951**, 2304.

[18] Andersen, E. K., *Acta Chem. Scand.*, **8**, 157 (1954).

[19] Fritz, J. S., *Anal. Chem.*, **24**, 306 (1952).

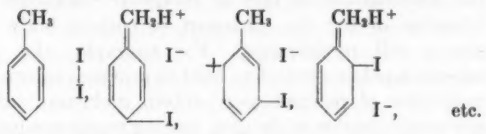
[20] Ferguson, L. N., "Electron Structures of Organic Molecules," pp. 52–69, 251–68.

[21] Ross, S. D., *J. Am. Chem. Soc.*, **76**, 74 (1954).

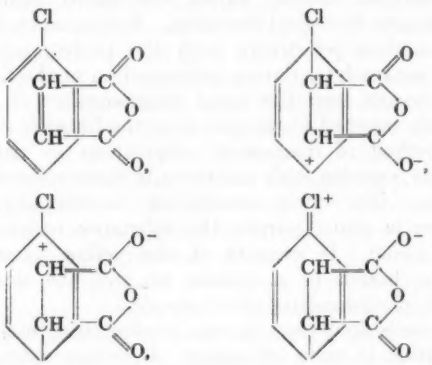
[22] See Andrews, L. J., and R. M. Keefer, *J. Am. Chem. Soc.*, **75**, 3776 (1953) for leads to many studies by these and other authors.

generally regarded as indicative of iodine-solvent interaction. An interesting color gradation is that of the alkyl halides with iodine, which can be taken as a qualitative measure of the relative tendency for iodine-alkyl balide association: alkyl iodide solutions of iodine are brown, alkyl bromide solutions are light brown, and alkyl chloride solutions are pink.

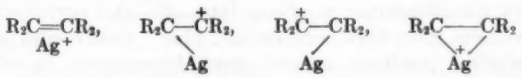
These complexes, which for convenience will be called pi complexes, are considered by many to owe their stability to the same type of forces operating in the solid molecular addition compounds discussed above. In resonance theory, such a structure can be described as a resonance hybrid of forms such as the following for a toluene-halogen complex, for example:



or for an aryl halide-maleic anhydride complex with the planes of the two molecules being parallel:

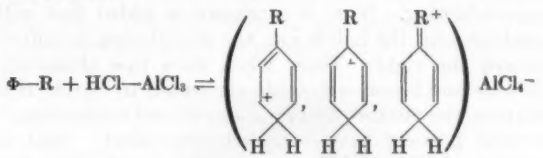


and for an alkene-silver ion complex:



From quantum mechanics, Mulliken[11] has shown that in an aromatic-halogen complex the halogen molecule is situated with its axis parallel to the plane of the aromatic nucleus and above the center of the nucleus, while in an aromatic-silver ion complex the silver ion lies above a carbon-carbon bond of the aromatic ring. This latter point has been verified by X-ray diffraction measurements on benzene-silver perchlorate crystals.[23]

Before we describe the pi complexes any further, let us point out another closely related complex and then discuss the two types together. Aromatic compounds react with, for example, HCl-$AlCl_3$ or HF-BF_3 mixtures to produce salts that ionize in nonaqueous, ion-supporting solvents like liquid hydrogen fluoride, sulfuric acid, or pyridine. The reaction can be represented by the equation



where Φ—R is an aromatic substance and the carbonium ion (in parentheses) is a resonance hybrid in which there is a covalent bond between the reagent and a definite carbon atom of the aromatic nucleus. Such an ion has been established as an intermediate in nitration where there is a nitro group in place of the additional hydrogen atom.[24] Although these carbonium ions, often called sigma complexes, are not molecular association products as are the pi complexes and the others previously discussed, the sigma complexes are so closely related to the pi complexes that it will be of interest to compare the two.

The first noticeable difference between pi and sigma complexes is their light absorption. The pi complexes absorb light with greater intensity and at slightly different wave lengths than do their components, but the change is primarily in the near ultraviolet.[25] On the other hand, the sigma complexes are colored and in some cases almost black.[26] As in the triphenylmethane-type dyes, the excited states of the sigma carbonium ions are greatly stabilized by resonance which decreases the energy difference between ground and excited states and permits light absorption in the visible.

The second distinguishing feature between pi and sigma complexes is the electrical conductivity of the sigma complexes, which is absent in the case of the pi complexes. The conductivity of the sigma complexes, of course, verifies that they are ionized in solution.

When aromatics are mixed with DCl-$AlCl_3$ or DF-BF_3, there is an exchange of deuterium for hydrogen. This satisfies expectation, for when the aromatic substance is recovered from its complex, either the C—H bond or

Electrophilic reagent		*Electron donor*	*Molecular complex*
A	+	B	⇄ A,B, $^-$A—B$^+$
Halogen Picric acid Oxygen Sulfur dioxide Tetranitromethane Quinone Maleic anhydride Silver ion Oxalyl chloride		Alkyl halide Alkene Aromatic hydrocarbon	

[23] Rundle, R. E., and J. H. Goring, *J. Am. Chem. Soc.*, **72**, 5337 (1950).

[24] Ingold, C. K., "Structure and Mechanism in Organic Chemistry," Cornell University Press, Ithaca, New York, **1953**, Chap. VI.

[25] Benesi, H. A., and J. H. Hildebrand, *J. Am. Chem. Soc.*, **71**, 2703 (1949).

[26] Akamatu, H., H. Inokuchi, and Y. Matsunaga, *Nature*, **173**, 168 (1954).

the C—D bond will break and when the C—H bond does break, the recovered aromatic will contain deuterium. No such exchange occurs in the pi complexes with DCl.[27]

An equilibrium apparently exists between an aromatic compound, its pi complex, and its sigma complex according to the equation

$$Ar + HX \rightleftharpoons Ar{\cdot}HX \rightleftharpoons ArH^+ + X^-$$

In media unfavorable to the existence of the ionic sigma complex, such as media of low dielectric constant, and in the absence of catalysts, only the easily formed pi complex is detectable. For instance, solutions of HCl in aromatic compounds are colorless and nonconducting. Now, if a reagent is added that will combine with the halide ion, the equilibrium is shifted toward the right. Thus, when salts like aluminum chloride and boron trifluoride are added to the Ar·HX mixture, the mixture becomes colored and conducting.[27] Several persons have reported this effect: that a mixture of HX + AlX_3 + Ar forms a red oil that is highly conducting but that does not form in the absence of any one of the three substances.[28] Also, liquid HF is a solvent of high dielectric constant so that aromatic substances can dissolve in it with the formation of a limited amount of sigma complex. This is evident, since hydrogen fluoride solutions of aromatic compounds have a conductivity and are visibly colored. When boron trifluoride is added to these solutions in increasing amounts, their conductivities and color depths increase until one mole-equivalent of boron trifluoride has been added, and then the conductivities level off, indicating that the equilibrium has been shifted almost completely to the right.[29]

Sulfuric acid too is a polar medium in which the ionic sigma complex is stable. Thus, aromatics undergo isotope exchange in D_2SO_4, verifying the existence of sigma complexes. Accordingly, C_6D_6 and other deutero aromatics have been prepared by mixing the aromatic compounds with D_2SO_4 or DCl-$AlCl_3$.[30]

Color alone should not be used as a criterion for distinguishing pi and sigma complexes. Isotope exchange or conductivity are better, although the conductivity will vary with the concentration of sigma complex. There are certain quantitative differences between pi and sigma complexes which need not be discussed here. Both types of complexes are valuable in facilitating our understanding of the nature of the electrons of the aromatic nucleus, something that has been a problem to organic chemists ever since the discovery of benzene.

The molecular addition compounds or clathrate compounds may have an important function not yet fully recognized. For illustration, it was mentioned above that the steroid desoxycholic acid forms clathrate compounds which dissolve without decomposition in alkali, and also it has long been known that purines such as caffeine form molecular addition compounds with many substances. Thus, aqueous solutions of sodium desoxyribonucleate and other purines dissolve many aromatic amines and polynuclear hydrocarbons (carcinogenic and noncarcinogenic).[31] This solubilizing effect raises this question: Is it possible that such a process, that is, intermolecular association, is responsible for the assimilation of fats or nonpolar chemicals in the intestine or for the transport of certain body agents across cell membranes? For example, the protein edestin has been found to hold in aqueous solution 5000 molecules of pentane per protein molecule. Proteins are very complex molecules, having many nonpolar and ionic spots along a single molecule, and various types of substances are adsorbed by proteins. Some of these combinations, usually called conjugated proteins,[32] have specific biological functions. For instance, Congo red combines selectively with the protein amyloid, which accumulates during inflammation of the liver or other organs, and the rapid disappearance of intravenously injected Congo red from the blood is used as one method of diagnosing amyloidosis in patients. Proteins combine with carotenes to form colored lipoproteins. The most interesting carotenoid-protein complex is visual purple, the substance required for night vision. It consists of the yellow carotenoid retinene bound to a protein to give the intensely colored, photosensitive visual purple.

In conclusion, then, it can be seen that molecular association is quite prevalent. It occurs extensively in solution and sometimes produces stable solid compounds. Some of the solid compounds, such as the urea complexes for purifying fats, oils, and petroleum products, have high commercial value. Other solid association products are of great importance in the laboratory; examples are the picrates for the isolation, purification, identification, and molecular weight determination of organic substances, and the urea complexes for the resolution of racemic mixtures. Their application to the resolution of amino acids is a possibility that could become a tremendous step in industry. The complexes in solution, such as the molecular addition compounds, and pi and sigma complexes, are currently of considerable theoretical interest and probably play important roles in biological systems.

[27] Brown, H. C., and J. D. Brady, *J. Am. Chem. Soc.*, **74**, 3570 (1952).

[28] Brown, H. C., and W. J. Wallace, *J. Am. Chem. Soc.*, **75**, 6268 (1953), and see references cited therein.

[29] Kilpatrick, M., and F. E. Luborsky, *ibid.*, **75**, 577 (1953).

[30] Klit, A., and A. Langseth, *Nature*, **135**, 956 (1935); C. K. Ingold, C. G. Raisin, and C. L. Wilson, *J. Chem. Soc.*, **1936**, 915.

[31] Booth, J., E. Boyland, and S. F. D. Orr, *J. Chem. Soc.*, **1954**, 598.

[32] Haurowitz, Felix, "Chemistry and Biology of Proteins," Academic Press Inc., New York, 1950, Chap. XI.

MARCELIN BERTHELOT AND INDUSTRY

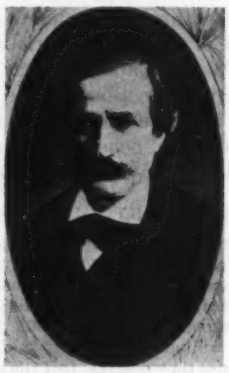
MARCEL DELÉPINE
Paris, France
(Translated by Ralph E. Oesper)

MUCH has been published concerning the life and achievements of Marcelin Berthelot.[1] In particular, the biography composed by Émile Jungfleisch, one of the most eminent of his collaborators, covers not less than 156 pages.[2] Numerous appreciations appeared during his lifetime and after, especially on the occasion of his scientific golden jubilee, the funeral at the Panthéon, the dedication of the monument (destroyed by the Nazis) before the Collège de France, and the dedication of the Maison de la Chimie, also known as the Centre Marcelin Berthelot.

However, the writers and orators have all missed one point, namely, the period in which Berthelot had some connection with chemical industry. To the writer's knowledge, no one has made mention of this.

The evidence of this activity is found in a laboratory notebook which shows that Berthelot worked from 1860 to 1863 for Menier at his chemical factory at Noisiel. At the time of Berthelot's death in 1907,[3] I was no longer his *préparateur* (assistant), a post that I had resigned in 1902, but I continued to frequent his laboratory at the Collège de France, where I had, as before, complete freedom to use the ample resources. I found this notebook abandoned in an old cupboard, and I do not believe that I have acted improperly in saving this scientific relic and studying its contents. Moreover, this brief phase in Berthelot's life had already been alluded to in print, since the *Moniteur scientifique de Quesneville* had carried a note in 1863 to the effect that Berthelot was consulting chemist to Menier.[4]

Although I thus knew that Berthelot had collaborated with Menier, I probably would have learned no more about this matter if I had not by chance met Gaston Menier (the son of the 1860 Menier) at a reception following a visit to the Menier chocolate factory at Noisiel in 1930. Knowing that I had been closely associated with Berthelot, Gaston Menier said, "I wager that you don't know that Berthelot worked for my father." I replied, "You will lose your bet; not only do I know that Berthelot was a collaborator of your father's, but



Marcelin Berthelot

I also know what he did, because I now have the notebook in which he entered the results of these studies." Gaston Menier requested the privilege of having this notable laboratory journal photographed. I then deposited the original in the archives of the Académie des Sciences along with a copy of the reproduction made on Gaston Menier's behalf.[5] Several copies were distributed by Menier. I was allowed to read the contract between Berthelot and Menier, Sr. It was couched in terms that were flattering and deferential to the scientist and correspondingly generous on the part of the manufacturer who had solicited his scientific cooperation.

To understand Menier's invitation and Berthelot's acceptance, it is necessary to recall that Menier conducted a factory at Noisiel for producing chemical and pharmaceutical materials, and that at this time Berthelot had just been appointed to the chair of organic chemistry created for him at the École de Pharmacie de Paris by an imperial decree of December 2, 1859. (He occupied this post for 17 years.) He was then already famous for his celebrated syntheses. It was quite natural, therefore, for him to accept an offer to collaborate in the preparation of chemical and pharmaceutical products.

[1] TRANSLATOR'S NOTE: Pierre-Eugène-Marcelin Berthelot (1827–1907), foremost French chemist of his time, became professor of organic chemistry in the École de Pharmacie in 1859 and in the Collège de France in 1865. In 1876 he was appointed inspector-general of education and was minister of public education in 1886–87. He served as foreign minister in 1895. His early studies established the theory of polyatomic alcohols, and he devoted much attention to explosives. His studies of the heat phenomena which produce and accompany chemical reactions were important in the founding of thermochemistry. He and his wife died within a few hours of each other; they are buried in the Panthéon.

[2] JUNGFLEISCH, E., *Bull. soc. chim. France*, (4), **13** (1913). (Special pagination followed by a list of the publications of Berthelot, covering more than 100 pages.)

[3] See THIS JOURNAL, **27**, 567 (1950).

[4] *Monit. sci. Docteur Quesneville*, **5**, 54 (1863). At this time, the name was written Ménier.

[5] *Compt. rend.*, **198**, 513 (1934).

The collaboration extended from October 26, 1860, to May 23, 1863. Berthelot went to the factory every eight days to plan the work and to advise those who did the actual experiments.

A page of the notebook is reproduced here; it is dated November 23, 1860, and shows Berthelot's handwriting. The subject matter deals with recommendations for the preparation of methyl oxalate and acetate, and also for the extraction of strychnine from nux vomica, together with a sketch of the apparatus.

On another page one reads, "Vendredi 31 Janvier 1862. Synthèse de l'alcool. Vendredi 7 Février 1862. Visite . . . Samedi 22. Visite à Paris des produits d'Exposition. . . ." This latter reference is to the World's Fair at London in 1862. Menier participated, and had requested Berthelot to get up a special exhibit of synthetic products. This was a novelty that attracted much attention, as is evident from the report composed by one of the most illustrious chemists of the last century, A. W. Hofmann, who was a member of the jury for the chemical section. Some extracts of this report are given here; certain historical portions, dealing with syntheses in general and of alcohol from ethylene in particular, have been omitted.

REPORTS BY THE JURIES

[Page 170] Attention will, in the next place, be claimed for some recent discoveries in organic chemistry, particularly valuable in a scientific point of view, as largely extending the domain, hitherto so restricted, of chemical synthesis, on the artificial construction of compound molecules; and, in connection with these discoveries it will become the Reporter's agreeable duty to record, with the praise they deserve, first the admirable synthetic processes devised by M. Berthelot, secondly the remarkable illustrations thereof contributed by M. Menier.

[Page 175] *M. Menier's Collection.* The early progress of organic chemistry is well known to have been accomplished almost exclusively by researches of an analytical or destructive character; it is only within the last few decades that the workers in this field of chemistry have become conscious of the synthetical or constructive powers of their science.... During the past few years synthetical operations in organic chemistry have become the order of the day, and none have cultivated this field with more ardor and success than the distinguished French chemist M. M. Berthelot, of whose processes the collection of chemical products exhibited by M. Menier...contains some splendid illustrations. Among these figure ample specimens of common alcohol, propylic alcohol, mustard oil, and formic acid, all synthetically produced.

The construction by M. Berthelot of formic acid from carbonic oxide and water (by means of hydrated potash)

$$CO + H_2O = CH_2O_2$$

is certainly one of the finest examples of synthetic chemistry.

The synthesis of alcohol from ethylene (olefiant gas) and water (by means of sulfuric acid)

$$C_2H_4 + H_2O = C_2H_6O$$

is equally elegant, and has perhaps attracted still more general attention, not only in the scientific world, as proved by the late important researches of M. Wurtz, but also among industrial speculators, as is well illustrated by the advertisements of M. Cotelle's Hydrocarbon Alcohol Company with which, some time ago, the columns of the Parisian press were teeming.

Hofmann's report shows not only that Menier made use of Berthelot's talents in the exhibition of his products, but that a corporation had already been organized to operate the synthetic formation of alcohol from ethylene. Obviously, the ethylene was not to be prepared from alcohol as it had been in the researches conducted by Berthelot.

It seems interesting to attempt to find out the nature of the Cotelle enterprise. Fortunately, all the required information can be gleaned from two articles published in the *Moniteur scientifique de Quesneville*. One of these was devoted to the London Exposition; the other, by a certain M. Mallet, dealt with the Cotelle proposal itself. The following paragraphs are from the first paper:[6]

Menier, of Paris, has prepared a very remarkable exhibit. His alkaloids from opium...have a whiteness and a beauty of crystallization that are the despair of the English manufacturers. His strychnine is amazing with respect to the size and the sharpness of the crystals; it might be supposed that this is a feat accomplished solely for the Exposition; actually what is put on the market is of the same high quality.[7]

... one novelty, that occupies a modest place in its glass case, has excited the lively interest of the specialists; it has raised the chemical work of the French to the level of the English from the scientific point of view. These are the synthetic products of Berthelot: alcohol made from illuminating gas, oil of mustard, oil of garlic, camphene, stearin, etc....produced completely in the laboratory at the will of the chemist. It is possible that coal may take the place of sugar and glucose as the raw material for the manufacture of alcohol; in fact, this has already been accomplished. Cotelle, a young employee of the Ponts et Chausées of Saint-Quentin, who for five years has applied himself to the realization of this idea, now produces two hectoliters of alcohol per day with gas retorts and oil as elements, with sulfuric acid playing the role of intermediary.... At some future time we will be able to give some details concerning the success of Cotelle.

These details are found in the second article published in the *Moniteur*,[4] entitled "The manufacture of alcohol by means of so-called illuminating gas." Berthelot disclosed his method of synthetically preparing alcohol on December 8, 1854. A month later, on January 15, 1855, Castex, of Puteaux, obtained a patent for a method of producing alcohol by means of gas from vegetable oils, fats, and all kinds of animal matter. Berthelot had made his alcohol from bicaburetted hydrogen (ethylene). After 53,000 strokes, he was able to produce sulfovinic (ethylsulfuric) acid by shaking this gas with sulfuric acid and mercury. On hydrolysis, this intermediate product yielded alcohol.

The author of the article added, "Up to the present M. Berthelot has not sought to derive any commercial advantage from this very curious experiment; he is satisfied to exhibit a liter of this alcohol, produced entirely by this method, at the London Exposition in the showcase of the Maison Menier, where he is consulting chemist."

What Berthelot did not attempt, Cotelle of Saint-Quentin sought to do by using the gas extracted from the oil for the manufacture of alcohol. A description of the process followed. In any case, it is *alleged* that the

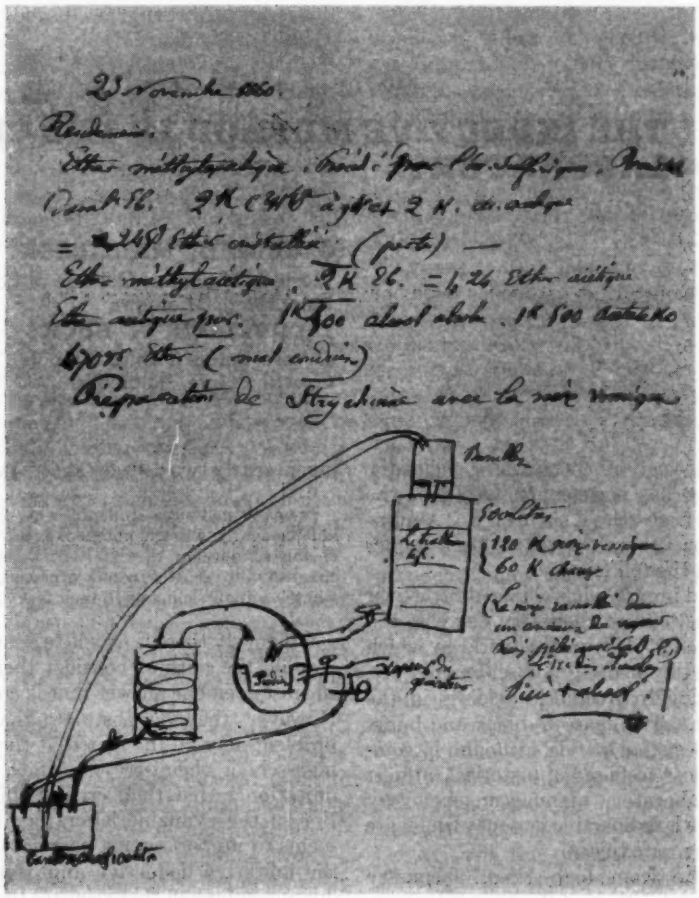
[6] *Monit. sci. Docteur Quesneville*, **4**, 433 (1862).

[7] There is little doubt that these specimens had been prepared by Berthelot.

French patent was sold for 500,000 francs and the English patent for 12 millions. (Since the dollar is now equivalent to about 400 francs, astronomical numbers result if these sums are translated into current exchange rates.) A corporation was organized at Saint-Quentin with a capital of 400,000 francs. All the shares were sold in an hour. The writer stated in conclusion, "The interested parties would do well, in any case, not to go to sleep in peace." Since there are no further reports of the outcome of this enterprise, it seems only too likely that this unfavorable prognostication proved very true. This attempt to go too rapidly from a laboratory experiment to technical practice deserves careful notice.

In 1901, on the occasion of the golden jubilee of the first publication by Berthelot, Leygues, the minister of public instruction, addressing the venerable scientist before a large audience gathered in the great auditorium of the Sorbonne, and using the florid language customary under such circumstances, said, "Abandoning all desire to collect the flood of gold gushing forth from your retorts, you asked as the reward for your efforts only the testimonial of your conscience."

It is strictly true that Berthelot exploited none of his discoveries. Though industrial use is now made of his synthesis of formic acid (also of oxalic acid), and of alcohols, by the union of water with such unsaturated hydrocarbons as ethylene, propylene, etc., and of persulfates by electrolysis, and so on, it must be remembered that years of work were needed before the processes were conducted on a profitable basis.



Page from Berthelot's Laboratory Journal

Preparation of methyl oxalate, methyl acetate, and strychnine from nux vomica

The "flood of gold" referred to by Leygues never poured over any of the stockholders of the Saint-Quentin company. Most inventions enter into a profitable cycle only after the technical experts have fitted them properly into association with other inventions. One of the aims of science and research is to give rise to and to further such useful applications. Although

Berthelot never derived monetary profit from his discoveries, he was thoroughly aware of the important role played by industrialists. At the opening of the International Congress of Applied Chemistry, which convened at Paris during the World's Fair of 1900, he defined their role in the world: "Are you not the incessant creators of the high value that is communicated to common materials by the intelligence and the labor of the human mind and hands?" But his own mental constitution was such that he could not long concern himself with profits derived from reactions and operations already known, and accordingly his efficient cooperation with the Menier industry was inevitably destined to be of short duration.

THE INDUCTIVE METHOD IN THE CHEMISTRY LABORATORY

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DURING the last few years at meetings of chemistry teachers much concern has been expressed about the laboratory program in high-school chemistry. A number of aspects of the laboratory have been under criticism, among them the following:

(1) The experimental program has changed little over the years although the field of chemistry has changed greatly. Almost the only place in the world in which oxygen is still prepared by heating potassium chlorate, or chlorine by oxidizing hydrochloric acid, is in the high-school laboratory. The charcoal block and borax bead are still used in tests for metals, although in commercial laboratories these tests are of historical interest only. Further, few laboratory manuals in chemistry suggest experiments with radioactive isotopes which are available and, with care, safe to use.

(2) Students emerge from high-school chemistry woefully naïve about implications of the term "elements of scientific method." Many still assume that *the* elements of *the* scientific method consist of five or six steps down which "rats inexorably and unvaryingly follow a Pied Piper."

(3) Students seem to be able to balance equations in a mechanical manner as though fitting together the pieces of a jig-saw puzzle. Yet the significance and implications of chemical reactions seem to evade them.

In other words, students do not seem to be able to think chemically. These criticisms have connotations similar to those found in an article written by Schlesinger[1] nearly two decades ago. In that article he stated:

> In many instances students, who, by their thoughtfully and independently written notebooks, have shown real interest in chemistry develop the habit of doing their experiments mechanically to get the result expected, rather than to observe what is actually going on in their test tubes.

Obviously there is no single answer to these problems. Nor do the authors consider themselves competent to suggest even one answer that is completely satisfactory. However, they do postulate that one reason for the problem may be the fact that the typical laboratory in high-school chemistry demands of the student mere imitation rather than critical thinking. An examination of the available laboratory manuals in chemistry seems to support such a postulate. The emphasis is on the imitative deductive approach rather than the inductive approach that requires the student to draw conclusions. Yet the former approach violates distinctly a major objective of the laboratory in chemistry, namely, that of teaching the student to solve problems related to the field of chemistry.

Of course it is easy to point out what is wrong, more difficult to suggest what may be right. However, the authors would like to offer a few suggestions that may help solve the problems that are implicit in the preceding discussion.

[1] SCHLESINGER, H. I., "The contribution of laboratory work to general education," J. CHEM. EDUC., 12, 524 (1935).

(1) *Dispense with Most Experiments Appearing in Laboratory Manuals.*

(*a*) "Problem: To Prepare and Study the Properties of Oxygen"; "Problem: To Etch Glass with Hydrofluoric Acid"; "Problem: To Study the Allotropic Forms of Sulfur"—so reads the typical laboratory manual in chemistry. Yet these are not real problems in any sense of the word. They are merely statements of sequences of activities in which the student will be forced more or less to participate. A problem exists only when a student finds himself in a situation in which he must seek answers in order to find a solution.

(*b*) Procedures. "Set up the apparatus as shown in the diagram"; "Thrust the glowing splint into the bottle"; "Mix thoroughly 5 g. of NaCl and 5 g. of MnO_2"; and so it goes endlessly, experiment after experiment! It is doubtful whether a chimpanzee would have much difficulty (or interest) in following such instructions; obviously the average chemistry student does not. Further, it is almost impossible to believe that merely following such directions will teach the student any elements of problem-solving or inspire much enthusiasm for increasing his efforts.

(*c*) Conclusions. "The properties of oxygen are ————"; namely, a blank to be filled in. However, if no real problem is posed how can conclusions, statements of the solution of the problem, ever emerge? Even if a problem were posed, the blank definitely limits possibilities for conclusions. The blank allows for little more than statements of findings or facts. There is little or no opportunity for the student to generalize.

In the last analysis the experiments amount to little more than "cookbook" manipulations, the results of which are to be written in blanks.

Of course these criticisms are not new. Hence one would be justified in suggesting that the authors should make some positive suggestions to remedy the weaknesses thus described. While they do not suggest that they have all or even one of the answers, the following pattern might help.

First, it should be made clear that the use of the deductive approach is useful with certain experiments. Second, the inductive approach cannot be used with one experiment and then forgotten until another suitable experiment appears. Actually the inductive approach should be used in all phases of the teaching process. Therefore, the following discussion will attempt to show how one lays the foundation for the inductive approach and then uses it with certain experiments.

(2) *Get Acquainted with Apparatus.* It is obvious that students cannot plan and set up their own experiments until they are aware of the potentialities and limitations of the available apparatus. Yet in most cases they learn of these characteristics incidentally by observing demonstrations or by slavishly carrying out experiments according to directions in the manual. Seldom are direct efforts made to point out to the student the potentialities of apparatus.

Hence it is suggested that during the first few days of the school year the student be shown the various pieces of apparatus and presented simple explanations and experiments that are designed specifically to illustrate their characteristics. Obviously this is necessary if students are later to plan experiments that will provide evidence toward the solution of some problem in chemistry.

An early experiment using the inductive approach might be one involving change of state. The teacher might proceed as follows:

"We've talked about the three states of matter, solid, liquid, and gaseous. The fact that substances can change from one state to another and back again is applied in a process called distillation. For example, certain materials may be dissolved in water. You may want to separate them from the water. This can be done by heating the water and dissolved material and then changing the gaseous water that escapes back into a liquid. I'd like you to work in small groups and with the apparatus in your desks set up equipment that will do the job. *Don't start it working until I look at it.* O.K., let's go!"

Obviously this introduction is sketchy and will be modified depending on the introduction to the study of states of matter, the type of student, etc. Also, some students always will be audience participants. However, most students will be forced to do some thinking—certainly more than if they were expected to set up equipment to match a diagram. Some may suggest that this exercise is simple. Of course it is. Yet the distillation of water appears in most chemistry laboratory manuals. It is less simple when students are required to figure out how to set it up.

After this introduction, another inductive-type experiment might follow. Perhaps one approach might be this:

"We've more or less had an introduction to the study of oxygen. However, we haven't discussed all of its characteristics. Perhaps it's about time we found out some more facts about it. You may recall that oxygen is a gas that is only slightly soluble in water and a little heavier than air. One makes it by mixing two solids and then heating them. Try to set up an apparatus that can be used to prepare and collect it. *Don't start it working until I have a chance to examine it.*"

Obviously this demands more thinking than "Set up the apparatus as shown in the diagram."

Later in the term when the students become acquainted with chemical equations the following approach may be used:

"Nitrogen dioxide is one of a number of oxides of nitrogen. It's heavier than air and soluble in water. It's prepared by heating copper in concentrated nitric acid. I'll give you an amount of solution that contains 20 grams of nitric acid. I want you to set up an apparatus in which this material can be prepared and collected safely. I want you also to figure out how many grams of copper shot you'll need."

Obviously from these examples of inductive experiments a teacher of chemistry can suggest many possible ramifications for many experiments. There are of course two "weaknesses."

(*a*) Will not these experiments take a "lot of time" and reduce the number of experiments a student can perform in a year?

The answer is "Yes!" However, if such a change will train the student to think, the time lost in imitation (which the psychology of learning has long since shown to be relatively valueless), is all on the "credit side of the ledger."

(*b*) Will not the course become tougher? The answer to this is "No!" The reduction in the number of experiments will give the students more time to *do* what chemistry teachers have paid lip service to for so long, namely, "to solve chemical problems." If this makes chemistry difficult, then it's about time chemistry teachers humbly agreed to forget this objective. This is one way to implement it. The repetition of processes carried out by chemists who do solve problems will not result in the infiltration of that skill into students.

(3) *Present the Laboratory Exercise Before the Topic is Discussed in Class.* Many chemistry teachers have been heard to say, "I prefer to give my laboratory work after I discuss the material in class. The students seem to know more about what they are doing." An excellent point! Unfortunately, however, the exercise is then no longer an information-gathering process. It is merely a device by which the students learn whether the teacher is correct, misinformed, or a liar!

If the chemistry laboratory in high school is designed to introduce the students to the techniques used by chemists, then it *must* be given prior to the classroom discussion. Do chemists only attack problems to which they know the answers? Obviously not! They attack problems to which they do *not* know the answers. Why then should not the student be expected to do likewise?

Perhaps the fundamental weakness in our thinking comes from the fallacy in the thinking of certain philosophers of science. They state that the word "science" comes from the Latin derivative *scio*, meaning, "I know." Logically it comes from *non scio*, namely, science begins when man does *not know!*

The implications for the laboratory in high-school chemistry are obvious.

(4) *Don't Rate the Students on the Accuracy of the Results of Their Laboratory Exercise.* While this suggestion of course needs to be tempered with discretion, one of the major weaknesses of the chemistry laboratory at both the high-school and college levels has been the demand for accuracy within rather narrow limits. Yet this occurs in spite of the following facts:

(*a*) Much of the equipment used by students is inferior and nonprecise, with flaws and weaknesses that frequently do not allow for accuracy within the limits demanded.

(*b*) The bane of all teachers of chemistry is the laboratory exercise or experiment that doesn't work! Yet the teachers accept this as a usual occurrence without self-recrimination.

(*c*) In commercial laboratories chemists repeat experiments many times in order to eliminate the possibilities of errors that can and do creep in. Yet chemistry teachers frequently demand of students accurate results the first time with equipment much inferior to that used by scientists who do not hope for such accuracy on the first occasion.

After once getting burned as a result of his errors, the student soon learns to be a clever swindler. He first learns, if possible, the correct answer, and works backward mathematically. He then inserts the data needed to produce the correct result, rather than data that result from his observation and experimentation.

A student learns the meaning of accuracy by analyzing his errors. Hence in the laboratory, in enhancing the inductive approach, the student should be given the correct answer after his answer has been turned in. At least a portion of his grade should be given on the basis of his ability to analyze his techniques and determine the possible causes of his error. In other words, what weaknesses in your procedures cause you to make the mistakes?

The writers are firmly convinced of two points:

(*a*) Such a system of chemistry laboratory will necessitate a reduction in the number of imitative exercises in order to allow for exercises in thinking. This is heartily to be commended.

(*b*) The interest of the student will be aroused because of the recognizable purpose in his efforts.

There are of course several cautions. The teacher must plan to use problems that are of importance to the student rather than those considered in some book as being "essential." Also, the demand for accuracy may be intensified as the course goes along—a demand based on the student's use of the knowledge developed by an analysis of his errors.

Perhaps further study of these problems, together with efforts to use some of the suggestions here, will make the chemistry laboratory more meaningful than heretofore. As emphasized by Schlesinger,[2] "...it is essential to keep in sight the major objectives—training in observation, in thought, and in action."

[2] *Op. cit.*, p. 528.

PHASE EQUILIBRIUM DIAGRAMS FOR MULTICOMPONENT SYSTEMS

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THE diagrammatic representation of phase equilibrium systems has receded into the background in recent years, owing, to a certain extent, to the greater interest in dynamic systems at the expense of systems in equilibrium. Also, with many of the problems met in practice, particularly in industrial fields, the number of components seldom can be restricted to the two or three which form the limit of experience for most graduates. Very often, when a more complex system is met, every endeavor is made to avoid detailed study, the difficulties of which are accentuated by methods described in textbooks (or omitted) for diagrammatic representation of results. The classic researches covering such complex systems as the salts of the Stassfurt salt beds have generally been described in terms of space models, which are always difficult to use quantitatively and to retain as a mental picture for comparative purposes. There have been numerous attempts to simplify this problem by the use of projections. But here again it requires an appreciable mental effort to relate these projections to the space model and obtain a real understanding of their meaning.

The purpose of this paper is to draw attention to the work of Jänecke (*1*) published during the earlier part of this century, which contains very useful ideas for the quantitative delineation of multicomponent systems. He put forward several methods of projection, but in the words of Purdon and Slater (*2*) "his presentation was not good and did not receive the merit it deserved, and in fact some fifteen years later (in 1921) Le Chatelier (*3*) put forward as new ideas, some of the ideas which Jänecke had introduced." While the author believes that Purdon and Slater in their book have rendered commendable service in bringing Jänecke's ideas before their readers, it is felt that even these authors have not done full justice to Jänecke. Rivett (*4*) also dismisses the Jänecke suggestions rather summarily, indicating preference for the more conventional pyramidal models. The author is of the opinion that the publication of Blasdale (*5*) provided a more easily understood account of Jänecke's schemes, and that extension of these can greatly facilitate the quantitative treatment of multicomponent phase equilibrium systems. The application of the Jänecke principle has found some favor for reciprocal salt-pair systems, and Findlay (*6*) has included this among other methods of treating four-component systems of this type. Ricci (*7*) has also drawn attention to Jänecke projections and very usefully discusses their applications with interesting examples. In the author's opinion, he likewise leaves the impression that their application is limited to the field of the reciprocal salt-pair type of system. In this paper it is submitted that there is a wider field to which this principle could be applied with advantage.

THREE-COMPONENT SYSTEMS

The more conventional method of delineating equilibrium conditions for three-component systems uses triangular coordinates with the sum of the three components equal to unity or some appropriate multiple such as 100. While this convention is frequently advantageous, the author feels that we have become too closely wedded to the idea and that more flexibility in methods of delineation is warranted for the wide range of conditions that is met in practice. It is considered that the use of rectangular coordinates for this purpose could be availed of more widely and the particular advantages of such a system of plotting employed in special cases. If rectangular coordinates are used, then the sum of two components is taken as unity and the third component identified as so much per unit sum of the other two. Jänecke (*1*), adopting this procedure, treated the solvent as the third component and represented it with a vertical axis, and 100 parts of dissolved solutes were represented along the horizontal base line of unit length. Objection was raised to this method of representation on the ground that pure solvent would be represented only at infinite distance from the horizontal axis. This objection was met by expressing solvent concentrations in terms of $100m/(100 + m)$ rather than by m, where m is the water associated with one part of solute mixture. Although this device yields a diagram which correctly expresses the relations of the different phases to each other, it gives a distorted picture of the actual concentrations concerned. The special cases for which the author believes this method of representation has definite advantages are those in which one component has a very low solubility, or in the case of alloy diagrams where it is desired to exaggerate the scale for one component to illustrate conditions in a small part of the conventional triangular diagram.

In Figure 1 an example is given in which sucrose and $CaSO_4$ are the solutes and water the solvent. The solubilities of $CaSO_4$ in sucrose are those determined by Stolle (*8*) in 1900, which serve to illustrate the point that the solute of low solubility—$CaSO_4$ in this case—may be represented on the vertical axis with advantage. Referring to Figure 1, for example, solution of composition represented by point E would, on evapora-

tion, change in composition along line *EF* in a direction away from the origin or point representing pure water. At point *F*, crystallization of $CaSO_4 \cdot 2H_2O$ will commence, and composition of liquid phase will change along the line *FB*. At point *B* sucrose will also crystallize, and a mixture of sucrose plus 640 p. p. m. of $CaSO_4$ as the dihydrate will continue to crystallize until evaporation is complete. Only solutions with a

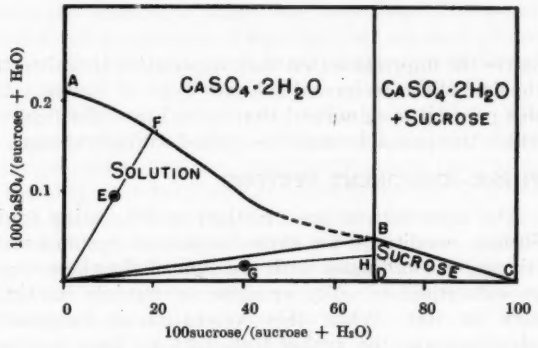

Figure 1. The System $CaSO_4$–Sucrose–H_2O

composition lying below the line *OB* will allow any sucrose free from $CaSO_4 \cdot 2H_2O$ to crystallize. For example, a solution with composition represented by point *G* would, on evaporation, change composition along line *GH*. At point *H* sucrose free from $CaSO_4$ would crystallize and continue to do so with the composition of the liquid phase changing from *H* to *B* when $CaSO_4 \cdot 2H_2O$ begins to crystallize. This diagram gives an indication of the difficulty of obtaining pure sucrose by crystallization if a second solute of low solubility is present. If it is desired to obtain useful

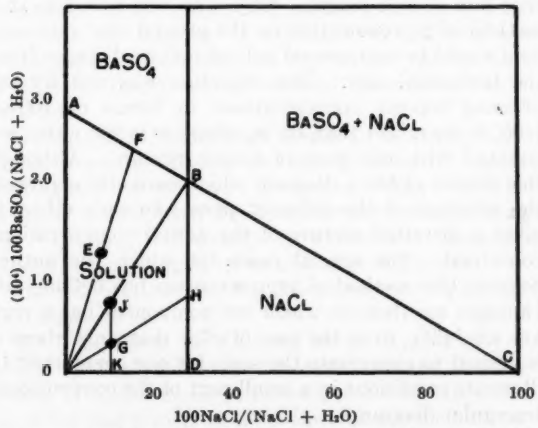

Figure 2. The System $BaSO_4$–NaCl–H_2O

proportions of pure sucrose it is necessary to remove the solute of low solubility by some means other than crystallization, *e. g.*, adsorption, and this of course is standard industrial practice in sugar refineries all over the world.

While this influence of low-solubility impurities may be fairly well known to many people as a matter of fact, the phase equilibrium relationships are not so often recognized as having such a bearing on the purification of chemicals. For example, in a sodium chloride solution from which sulfate has been removed by precipitation as $BaSO_4$ it is impossible to crystallize pure sodium chloride free from $BaSO_4$. The reason for this may be clearly illustrated in a phase-equilibrium diagram using vertical and horizontal coordinates in the manner just described, with $BaSO_4$ represented on the yertical axis; this is illustrated in Figure 2. The composition of any solution prepared in this manner will lie somewhere along *AB*, as it will be saturated with $BaSO_4$, and $BaSO_4$ will crystallize on evaporation, with composition of the liquid phase moving along to *B*, at which stage NaCl will begin to crystallize but will do so with approximately 80 p. p. m. of $BaSO_4$. If the initial solution is treated for removal of $BaSO_4$ by an adsorption process, enough must be removed to bring the composition of the liquid phase into the area *OBD* if any crystalline NaCl free from $BaSO_4$ is to be obtained. For example, if the purified solution composition may be represented by point *G*, then the proportion of NaCl which may be crystallized free from $BaSO_4$ will be represented by *JG/JK*. On evaporation, the composition of the solution will move along *GH*, when pure NaCl will commence to crystallize upon evaporation, the composition of the solution changing along *JB* until the "impure" NaCl begins to crystallize. Any point in the area *OAB* will give free $BaSO_4$ after sufficient evaporation. For example, the solution of composition represented by point *E* will change in composition until it reaches *F*, at which stage $BaSO_4$ will crystallize and the composition of the liquid phase will change along line *FB*, and when it reaches *B* then NaCl with 80 p. p. m. of $BaSO_4$ will crystallize.

In the preparation of the diagram no allowance has been made for possible conditions of supersaturation, or for the possible influence of one salt on the solubility of the other. It is also recognized that this system is really that of a reciprocal salt pair in which four different solid phases might be expected from the various possible combinations of the ions. However, for the purpose of examining the conditions for preparing a pure crystalline NaCl free from sulfate, the diagram as illustrated is considered to show the requirements with the maximum of clarity.

In both Figures 1 and 2 it is recognized that pure material represented by the vertical axis would be at an infinite distance from the horizontal axis. This, however, is considered to be a minor deficiency compared with the advantages in use of this system for purposes such as those just described.

In the case of alloy systems the great difficulties experienced in obtaining metals of really high degrees of purity may be illustrated in similar types of diagrams, where the element of low concentration is represented on the vertical axis. Similarly, in silicate systems or other systems in which solid solutions are prominent,

coordinates of this nature may be used with advantage to draw attention to specific conditions occurring where one compound is present in small concentration.

FOUR-COMPONENT SYSTEMS

Blasdale (*5*) has described clearly the Jänecke method of representing four-component systems in which the percentages of three solutes are referred to the sides of an equilateral triangle and the solvent associated with 100 parts of solute to perpendiculars to the plane of the triangle. The author has found this system to be very useful in practice and relatively easily comprehended, particularly if pictured as a projection of the surface of a vertical prism on the base. Perhaps this is more easily understood than the picture given by Purdon and Slater (*2*), who describe the Jänecke projection as assuming that a shadow is cast from a point of light situated at the apex of a pyramid.

The projection of the surface of the vertical prism on its base gives a triangle with contour lines representing changes in solvent quantities. Similar criticism may be made of these diagrams as for those with three components where solvent is the vertical axis and pure solvent is at infinite distance from the horizontal axis. For the four-component system represented by a vertical triangular prism with solvent as the vertical axis, pure solvent would be at infinite distance from the triangular base. The contour system of marking solvent concentrations is, however, very convenient, and most of the usual calculations for a three-component system may be carried out with this type of diagram. In Figure 3 is reproduced the diagram for the system $BaCl_2$–$CuCl_2$–KCl–H_2O prepared and published by Blasdale (*5*) from results obtained by Schreinemakers and de Baat (*9*). The author (*10*) is at present preparing diagrams for the publication of results obtained by him for four-component systems of sugars, inorganic salts, and water. This method of representation has proved particularly helpful in the preparation of preliminary diagrams from which synthetic solutions are prepared, and useful indications given of the conditions likely to be obtained in the final system.

Four-component alloy or silicate systems may be represented by triangular diagrams with the concentration of the fourth component shown as contours and represented as a concentration per unit of the sum of the other three components. Where such systems are complex it is necessary to use discretion as to which of the components is represented as the fourth component. It may even be necessary to use two or even more diagrams to illustrate a system completely, but such would be the case even with projections from space models such as the tetrahedron. The author considers that the contour system may be comprehended more easily and may be more amenable to quantitative treatment.

Where solutions are being dealt with there may well be cases in which one of the solutes could, with advantage, be taken as the fourth component. This would more likely be the case where one of the solutes has a very low solubility, as was true of the three-component systems just described.

It is also possible, and sometimes both convenient and desirable, to employ rectangular coordinates similar to the example of Figures 1 and 2. However, in a four-component system two components would be represented on the vertical axis with the quantitative relationship indicated as so much per unit sum of the first two components. The fourth component would then be represented by contour lines on the diagram and quantitatively referred to as so much per unit sum of the first two components.

FIVE-COMPONENT SYSTEMS

The contour system of delineation may also be used for five-component systems. In the case of solutions with four solutes, one of these solutes may be selected as a component to be illustrated with a second set of contours and concentrations referred to as so much per unit sum of the other three solutes, which are themselves

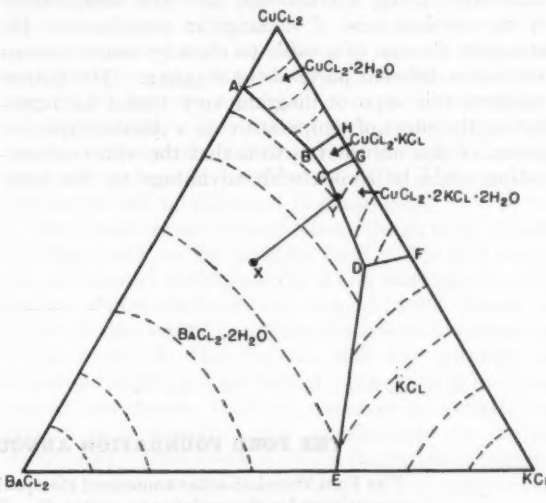

Figure 3. The System $BaCl_2$–$CuCl_2$–KCl–H_2O

represented at the corners of an equilateral triangle. Two or more diagrams will be necessary to represent conditions adequately. The author is at present working on a system which will be expressed in a diagram of this nature.

MORE COMPLEX SYSTEMS

The contour system may be extended to even more complex systems by the exercise of a certain amount of ingenuity and use of multiple diagrams, and such a method is considered to be more useful than space models and projections, which are difficult to visualize.

Reciprocal salt pairs in which six solid phases may appear offer a problem of a special nature, but may be treated by the contour system with triangular basic coordinates and a multiple set of contour lines. However, this about reaches the limit of complexity which may be represented with any reasonable degree of

satisfaction. Systems in which there are three bases and three acid radicals offer the possibilities of nine individual solids aside from the possibility of double-salt formations, and are extremely difficult to represent graphically by whatever system may be adopted. Fortunately, with most of these very complex systems it is seldom necessary to work out the full diagram, as interest may be concentrated over relatively small concentration ranges. The original paper of Jänecke (*1*) is well worth while studying before dealing with complex problems of this nature, as he has some useful diagrams of complex salt systems.

TEMPERATURE AND PRESSURE CONSIDERATIONS

Blasdale (*5*) draws attention to what he considers a more important use of Jänecke's scheme in representing the effect of temperature upon a system. He illustrates the effect of temperature on the system Na_2SO_4–$MgSO_4$–H_2O between $-10°C.$ and $+100°C.$, in which the relative proportions of Na_2SO_4 and $MgSO_4$ are represented along a horizontal axis and temperature on the vertical axis of rectangular coordinates. He advocates the use of a table to identify water concentrations in different parts of the diagram. The author considers this type of diagram very useful for representing the effect of temperature on a three-component system of this nature, but feels that the water concentration could be shown with advantage on the same diagram with the use of a contour system of representation, thus eliminating the necessity for using a chart as an additional aid to interpretation.

If pressure variation is important to the system there is no reason why this could not be represented on the vertical axis in a diagram of this nature, instead of temperature, as Jänecke himself has done. The simultaneous representation of changes in both temperature and pressure on the same diagram for a three-component system does, however, present some difficulties, even if triangular coordinates are used.

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THE FORD FOUNDATION ANNOUNCES FELLOWSHIP COMPETITION

The Ford Foundation has announced the opening of its Foreign Study and Research Fellowship competition for the academic year 1955–56. The awards, which will be made in April and May, 1955, are for study and research dealing with three areas: Africa, Asia and the Near East, and Soviet Russia and Eastern Europe.

The purpose of the awards is to increase the number of Americans professionally competent to interpret the cultures, histories, and current problems of these foreign areas.

This is the fourth series of fellowships offered under the Foundation's Foreign Study and Research Fellowship Program since it was initiated in 1952. In the three previous years fellowships were awarded to a total of 313 young men and women, enabling them to begin or continue research and training pertaining to foreign countries.

The fellowships, covering from one to three years of postgraduate work either in the United States or abroad, are designed to provide training for persons at a variety of academic and professional levels. Students just beginning their graduate work may apply, as well as advanced scholars. A limited number of awards will be made to persons of recognized competence outside of academic fields.

The present competition is open to United States citizens and to aliens permanently residing in the United States who can give substantial evidence of their intention to become citizens. Applicants should not ordinarily be over 40 years of age, and age limits of 30 and 35 apply to certain of the Soviet and East European Area fellowships.

Details and application forms may be obtained from The Ford Foundation, Foreign Study and Research Fellowship Program, 477 Madison Avenue, New York 22, New York. The deadline for filing applications is January 7, 1955.

PREDICTION OF THERMODYNAMIC DATA

A Problem in Chemical Engineering

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ONE of the great differences between problems given students in engineering courses and those encountered by practicing engineers is the amount of technical data available for the problem's solution. For problems assigned in school, the data required are often contained in the student's textbook or handbook. On some occasions the student must go to a technical reference to acquire the necessary data. To save the student a long search of the literature, the reference is often given to him. Regardless of the actual source of data, they are available.

Problems of this type are assigned so that the student can use some basic principle in their solution. In school one attempts to teach only the fundamentals. In selecting problems for which the required data have been determined, an erroneous impression is created in the student's mind, for he is led to believe that the data needed for the solution of all problems are accessible. On the other hand, for many of the problems confronting the engineer in industry the technical data are not available. Moreover, the engineer may have neither the time nor the money to determine the facts experimentally. In such cases he must try to predict the data, which may be of many different types.

One purpose of this paper is to present a type of problem that can be given to students in chemical engineering thermodynamics so that they can appreciate to some degree one of the problems of the engineer in industry. A second purpose is to illustrate some of the important points in the problem's solution, this being done by means of a specific example.

The problem recommended here for assignment is the prediction of thermodynamic properties for a specific compound. This would include the determination of enthalpy and entropy as functions of temperature and pressure up to the critical pressure and temperature. These properties can be evaluated for the saturated liquid and vapor states and the superheated vapor state.

The compound cyclohexane has been chosen for the purpose of demonstrating a typical solution. The complete solution will not be offered—only as much as is necessary to clearly define the method. The student's first step is a search of the literature to determine the available data on cyclohexane. A review of *Chemical Abstracts* serves as a good starting point. The search will reveal that the enthalpy and entropy data for the substance in the ideal gas state are available in API Project 44 (*1*), as are vapor pressure data, the critical constants, and two latent heat of vaporization values. With the exception of the latent heat information, these data represent the minimum required for the problem's solution. Other references (*2*, *4*, *9*, *14*, *15*) report data similar to those compiled in API-44, and do not aid further in the solution of the problem. Auerbach, Sage, and Lacey (*3*) reported the isobaric heat capacity of cyclohexane at the bubble point. These data are useful, as will be shown later. The only volumetric data available are those of Nagornov and Rotinjanz (*11*), which were measured at low pressure and temperature and are not of use in the solution of the problem. The student, being confronted with this lack of experimental data, must then seek methods to predict thermodynamic data. These methods of prediction will be discussed in detail later.

The literature survey completed, the student is ready to begin work on the problem itself. The first step is the selection of the datum plane and the basis of calculation. Since engineers use English units almost exclusively, the logical basis for calculations is a pound of cyclohexane. It then follows that the enthalpy be expressed in B.t.u.'s per pound, entropy in B.t.u.'s per pound per degree Rankine, pressure in pounds per square inch absolute, and temperature in degrees Fahrenheit. The selection of the datum plane requires a little judgment and a knowledge of the temperature and pressure range over which the compound is most frequently used. The datum plane is that point where the enthalpy and entropy of a substance in a particular state are set equal to definite values. These values are usually zero. For instance, in the API-44 the datum plane temperature is 0°R. and the prescribed state of the substance is that of an ideal gas with unit (14.7 p. s. i.) fugacity. Under these conditions the entropy and enthalpy of the substance are set equal to zero. The student could easily select this as the datum plane for his calculation, but this often leads to negative values for the enthalpy and entropy of the substance in the liquid state at ordinary temperatures. Since some people find it awkward to handle negative values, it is best to avoid them unless there is some compelling reason for not doing so. In the student problem such a reason is not present, hence the datum plane should be chosen to avoid negative values for these thermodynamic properties. In the case of cyclohexane the liquid under its own vapor pressure at 44.1°F. is a

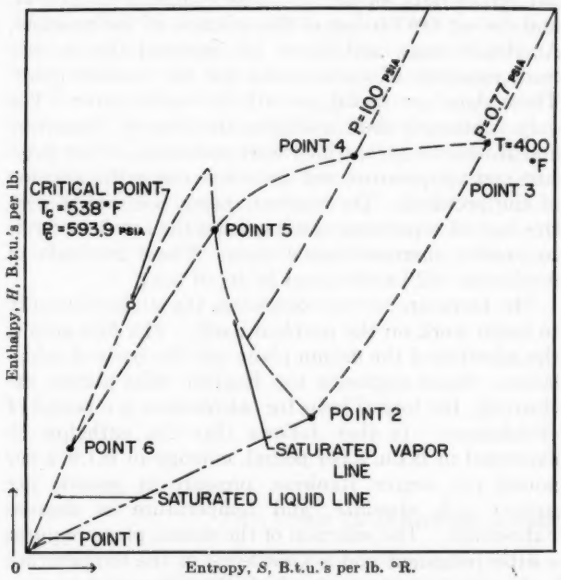
suitable datum plane, this temperature being the fusion temperature. The entropy and enthalpy of cyclohexane are set equal to zero at these conditions, point 1 on the figure.

Having established the values of the entropy and enthalpy in the liquid at 44.1°F., it is possible to determine these quantities for the vapor in equilibrium with the liquid if the latent heat of vaporization is known. Equations 1 and 2 show the relationship between these enthalpies and entropies.

$$H_{\text{sat. liquid}} + L_v = H_{\text{sat. vapor}} \tag{1}$$

$$S_{\text{sat. liquid}} + \frac{L_v}{T} = S_{\text{sat. vapor}} \tag{2}$$

The latent heat of vaporization for cyclohexane is not available at the temperature in question, so some means must be used to predict it. At this low pressure, 0.77



Typical Enthalpy Versus Entropy Diagram

p. s. i. a., the gas phase behaves practically ideally and the volume of the liquid phase is negligible in comparison to that of the gas phase; therefore the Clausius-Clapeyron equation, equation 3, can be used to calculate the latent heat.

$$\frac{dP}{dT} = \frac{ML_vP}{RT^2} \tag{3}$$

The thermodynamic properties of the saturated vapor at 44.1°F. are then determined and point 2 located on the figure. Also, it is now possible to convert all the API-44 thermodynamic data to the datum plane used in this problem. This enables the student to draw in completely the 0.77 p. s. i. a. isobar. Point 3, T = 400°F., represents one point on this line.

It now becomes necessary to determine the changes in enthalpy and entropy with pressure under isothermal conditions. To calculate these changes the chemical engineer has four possible correlations from which to choose, namely, Watson and Smith (*17*), Edmister (*5*, *6*), Hougen and Watson (*8*), and York and Weber (*18*). The last cited reference deals only with conditions above the critical temperature; therefore its use is limited and will not be discussed. Hougen and Watson use a combination of the methods employed by Edmister and York and Weber, so a discussion of it, likewise, will be omitted. The correlation of Watson and Smith is based on the relationship

$$PV = CRT \text{ for one mole of gas} \tag{4}$$

where C is the compressibility factor and a function of the reduced pressure and reduced temperature. This is an application of the law of corresponding states, familiar to all chemists and chemical engineers. The equation of state was used in the thermodynamic relationship for the change of enthalpy and entropy at constant temperature. For the case of enthalpy the equation of state was substituted in the relationship

$$\left(\frac{\partial H}{\partial P}\right)_T = V - T\left(\frac{\partial V}{\partial T}\right)_P \tag{5}$$

with the result that the expression for the change of enthalpy with pressure is:

$$\frac{H^\circ - H}{T} = RT_R^2 \int_0^{P_R} \left(\frac{\partial C}{\partial T_R}\right)_{P_R} d \ln P_R \tag{6}$$

The change in enthalpy divided by the absolute temperature, the left-hand side of the equation, was then plotted as a function of the reduced temperature and pressure. The change in entropy with pressure at constant temperature was determined by the same general procedure with one major variation, this being due to the fact that the change in entropy with pressure at constant temperature for an ideal gas is not zero, as is the case with enthalpy, but is expressed by the following equation:

$$(S^\circ_{P_2} - S^\circ_{P_1})_T = -R \ln \frac{P_2}{P_1} \tag{7}$$

Being cognizant of this fact, Smith and Watson present a chart of entropy differences between the ideal gas and a real gas, both at the same temperature and pressure, plotted as a function of the reduced temperature and pressure.

Edmister's correlation (*5*, *6*) makes use of the reduced equation of state:

$$\frac{V}{\alpha_c} = \left(\frac{RT_c}{P_c\alpha_c}\right)\left(\frac{T_R}{P_R}\right) - \alpha_R \tag{8}$$

where α_R is a general function of the reduced temperature and pressure. This is the expression:

$$\alpha = \frac{RT}{P} - V \tag{9}$$

written in reduced form. Using the reduced equation of state and the proper thermodynamic relationships, Edmister constructs charts similar in their general nature to those of Watson and Smith, but differing in detail because of the selection of a different equation of

state. The student can learn a great deal of basic thermodynamics by studying, in detail, the methods used in the construction of the charts cited above.

The use of a particular correlation for the evaluation of enthalpy and entropy changes with pressure at constant temperature is easy, and in this case only requires a knowledge of the critical properties of cyclohexane. For example, point 3 on the figure is fixed at the temperature of 400°F. and 0.77 p. s. i. a. To calculate the enthalpy and entropy changes from point 3 to 400°F. and 100 p. s. i. a., point 4, the student need only calculate the reduced temperature and pressure at the new condition and obtain from the appropriate chart the change in enthalpy and the entropy resulting from deviation from ideal conditions. Since the enthalpy change is entirely due to the departure from ideal behavior, the figure obtained from the correlation may be used directly. The total entropy change, however, is made up of the sum of two quantities, the change for an ideal gas between the two pressures and the change due to the deviation from ideality, mathematically:

$$(\Delta S)_T = -R \ln \frac{P_2}{P_1} - (S^{\circ}_{P_2} - S_{P_1}) \qquad (10)$$

The amount of the second term is determined from the appropriate correlation. These calculations can be repeated for a number of pressures at the constant temperature of 400°F., until the vapor pressure, 206.55 p. s. i. a. in this case, is reached. This gives a second point on the saturated vapor line and is represented by point 5 on the figure. This procedure can be repeated for a number of temperatures and the entire thermodynamic network established in the superheated and saturated vapor regions.

To establish the saturated liquid curve the student has, theoretically, the choice of one of two methods. One of these methods requires latent heat of vaporization data and simply is the subtraction of the enthalpy or entropy change during vaporization from their respective values in the saturated vapor state. The other method requires isobaric heat capacity data at the bubble point for the substance. These heat capacity data are available for cyclohexane over the temperature range from 80 to 200°F. (*3*). The more usual case is that these data are not in the literature. When they are available a good check on the calculations is possible, since enthalpy and entropy changes between two states can be evaluated along two different paths. These changes are independent of the path.

The more common method for determining the thermodynamic properties in the saturated liquid state is by use of the first method given, using the latent heat of vaporization. This requires a knowledge of the latent heat at numerous temperatures, which is seldom obtainable in the literature. In the case of cyclohexane four values of the latent heat are available, one at the normal boiling point and three at lower temperatures (*1, 9*). There are a number of methods that can be used to predict the latent heat. Actually it would be worth while for the student to calculate the latent heat by a number of different methods, to permit a comparison of the results. Some of the correlations more frequently used are those of Othmer (*12, 13*), Gordon (*7*), Su (*16*), and Meissner (*10*). To go into the detail of these methods is beyond the scope of this paper. It may be pointed out that some of them make use of the Clapeyron relationship, with various assumptions, and the law of corresponding states.

Returning to the specific example of cyclohexane, the student can predict the latent heat of vaporization at 400°F. and from there can readily locate by the use of equations 1 and 2 the proper point on the saturated liquid line, labeled point 6 on the figure. The procedure can be repeated for a number of temperatures, thereby completely defining the liquid line. With this step the diagram is finished.

In this paper a problem in the prediction of thermodynamic data has been recommended for students in chemical engineering thermodynamics. The specific method of solution has been indicated for cyclohexane. The other compounds that can be used in this type of problem are *n*-hexane, diisopropyl, neohexane, isoheptane, diisobutyl, *n*-octane, and toluene, to name only a few of the possibilities.

Nomenclature

C	=	Compressibility factor, defined by equation 4
H	=	Enthalpy, B.t.u.'s per lb.
L_v	=	Latent heat of vaporization, B.t.u.'s per lb.
M	=	Molecular weight
P	=	Pressure, lb. per in.^{2} absolute
R	=	Gas law constant, 10.73 lb. per sq. in. per ft.^{3} per lb. mole per °R.
S	=	Entropy, B.t.u.'s per lb. per °R.
T	=	Temperature, °R.
V	=	Molal volume, ft.^{3} per lb. mole
α	=	Residual volume, defined by equation 9

Subscripts:

c = critical condition
R = reduced condition

Superscripts:

o = ideal gas state

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NEW SALTS OF PARA-XYLENE SULFONIC ACID

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THIS was a senior research project carried out under the direction of and with the aid of the chairman of the chemistry department. The experience was an invaluable one from the initial library search to the thrill of new discoveries and the drafting of the final report. The students who did this work will be prepared to use their initiative and to cope with new problems when they leave college and enter industry.

A thorough library search showed that only a very few salts of para-xylene sulfonic acid had been prepared, namely the sodium, barium, potassium, and beryllium salts. The recent availability of pure para-xylene[1] at moderate price, the ease with which it can be sulfonated, and the high yield of the acid were factors in the attempt to prepare new salts. A simple method of preparing the acid was presented by Weber in THIS JOURNAL.[2] By modifying Weber's method the time was considerably shortened.

Several methods of preparing the salts were tried. The direct reaction of the acid with salts of the desired ion proved to be the best. Eighteen salts were attempted, of which 16 were successful. The parent compound crystallized in long, colorless needles. The salts formed three kinds of crystals: needles similar to the acid; long, flat crystals with pyramid ends; and rectangles with cut-off corners. The ammonium salt formed the largest and most perfect crystals. Solu-

[1] Pure para-xylene can be obtained from Eastman Distillation Products, Rochester, N. Y.

[2] WEBER, J. E., J. CHEM. EDUC., **27**, 384 (1950).

Characteristics of the Salts

Salt	*Reagent*	*Crystal form & color*	*Solubility*		
			Water	*Alcohol*	*Ether*
		Needles			
Cadmium	$CdCl_2$	Fine, white	s.	i.	i.
Cobalt	$Co(NO_3)_2$	Long, pink	s.	s.	v. sl. s.
Aluminum	$AlCl_3$	Extremely fine, white	s.	s.	s.
Magnesium	$MgCl_2$	Fine, white	s.	v. sl. s.	v. sl. s.
Manganous	$MnCl_2$	Fine, faint pink	s.	s.	v. sl. s.
Nickelous	$NiSO_4$	Long, light green	s.	s.	v. sl. s.
Zinc	$ZnCl_2$	Long, white	s.	s.	v. sl. s.
		Long, flat rectangles, pyramid ends			
Copper	$CuCl_2$	Light blue, glassy	s.	s.	v. sl. s.
Antimony	$SbCl_3$	Colorless, glassy	s.	s.	v. sl. s.
Ferric	$FeCl_3$	Bright yellow, glassy	s.	s.	v. sl. s.
Lead	$Pb(NO_3)_2$	Colorless, glassy	s.	v. sl. s.	v. sl. s.
Strontium	$SrCl_2$	Colorless, glassy	s.	sl. s.	v. sl. s.
Zirconium	$ZrCl_4$	Colorless, glassy	s.	s.	i.
		Rectangles, corners off			
Ammonium	NH_4OH	Clear, colorless	s.	sl. s.	v. sl. s.
Bismuth	$BiCl_3$	Clear, colorless	s.	s.	i.
Stannous	$SnCl_2$	Clear, colorless	s.	s.	i.

bility was tested in water, alcohol, and ether according to Kamm's method,[3] one part to 30 parts or 0.1 g. salt to 3 ml. solvent.

PROCEDURE FOR PARA-XYLENE SULFONIC ACID

Para-xylene was placed in a short-necked, round-bottomed flask and a little more than twice as much concentrated sulfuric acid was added with swirling. The mixture was heated only until it became warm. With continued, vigorous swirling the product was homogeneous in 10 to 15 minutes. Reaction was complete if there was no separation when swirling was discontinued. Cold water, slightly less in amount then the para-xylene, was added while still hot and the contents poured at once into a beaker, as there was immediate precipitation. After filtering with suction it was recrystallized from as little water as possible.

PROCEDURE FOR THE SALTS

The sulfonic acid was dissolved in as little water as possible to make a clear solution. The salt of the desired ion was dissolved in the same amount of water to make a saturated solution and the two combined. In most cases there was immediate precipitation. Heat was applied and just enough water was added to make a clear solution, which was allowed to cool and crystallize slowly. The salts were filtered by suction and recrystallized from as little water as possible. Most of the salts crystallized quickly, a few took several days, and some stubborn ones were evaporated with an infrared lamp.

[3] KAMM, O., "Qualitative Organic Analysis," 2nd ed., John Wiley & Sons, Inc., New York, 1945, pp. 138, 139.

A SIMPLE MELTING-POINT APPARATUS

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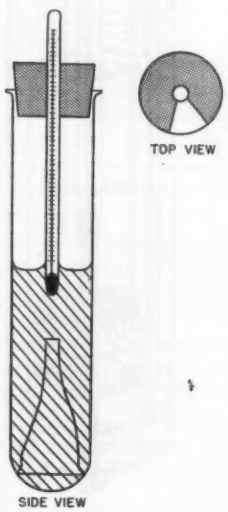
THE melting-point apparatus described in this paper is distinguished by extreme simplicity of construction and negligible cost. It has been tested by the writer and his students for years and compares most favorably with more elaborate and expensive apparatus.

A 25 × 150-mm. Pyrex test tube and a 15 × 85-mm. soft-glass test tube are needed to construct the apparatus. The small test tube is softened in a flame about 2 to 4 cm. from its open end and narrowed to a diameter of about 2 to 4 mm. It is not at all necessary to do this carefully; as will be explained below, the apparatus works better if the narrowing is not accurately centered.

The small test tube is now cut off at the narrow point and its funnel-shaped top placed inside the large test tube upside down, *i. e.*, so that the mouth of the funnel rests on the bottom of the large test tube and the tip of the funnel points upward. Bath fluid is then poured into the large test tube until the fluid level is about 3 cm. above the tip of the funnel, and the test tube is closed with a cork stopper. A hole is bored in the stopper to hold a thermometer, and a sector is cut out in its side for venting and for making the whole scale of the thermometer visible. (See the figure.)

A sample of the substance to be tested is placed in a melting-point capillary and attached to the thermometer in the usual way, and the position of the thermometer adjusted so that its entire bulb is just below the surface of the bath fluid. If now a microburner is placed under the test tube, most of its heat will go into that part of the fluid which is inside the inverted funnel. The fluid will therefore expand and rise from the funnel,



quite fast and with considerable turbulence, owing to the narrowness of the opening, especially if the warm fluid rises near the walls of the test tube rather than in the middle. This is the reason why a funnel with a poorly centered tip is preferred.

The eddy currents which are thus set up mix the upper part of the bath fluid effectively, no matter whether heating is fast or slow. In all cases the thermometer and the sample are heated evenly, and melting points of excellent reproducibility are obtained.

A NEW MELTING-POINT APPARATUS[1]

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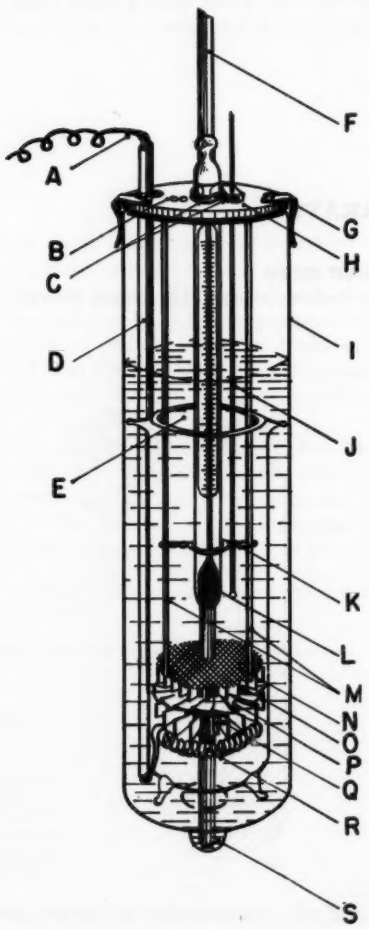
THE author has used the modified Hershberg melting-point apparatus[2, 3] for a few years. In this apparatus the silicone fluid, after having been heated by the internal heating coil and mixed by the stirrer, has to travel a long way before it comes in contact with the bulb of the Anschutz thermometer and the bottom of the melting-point tube containing the sample of the substance under investigation. The heated liquid must be uniformly mixed by the stirrer but on its further travel it comes in contact with the exposed walls of the apparatus, making the outer surface of the moving column of the liquid colder than the inner bulk. Further on, when the liquid column makes a turn down toward the thermometer bulb, the liquid cooled by the exposed walls tends to mix with the inner liquid. At this stage one can actually observe streaks of silicone fluid of different refractive indexes, presumably the result of the difference in their temperature, moving down toward the region of the thermometer bulb. Although the temperature difference between these streaks would be difficult to determine and may not be very large, their presence indicates that the temperature of that cross section of the column of silicone oil at the level of the bulb is not uniform.

In order to overcome this difficulty a new type of melting-point apparatus was constructed; it is shown in the diagram.

It consists of an outer vessel *I* made of Pyrex tubing of 45-mm. outside diameter, $6\frac{1}{2}$ inches high, open at the upper end, and closed at the lower. The closed bottom end has a small well *S* which is about half an inch deep and has an outside diameter of 12 mm. This well is located at the center of the bottom of the outer vessel and serves as a bearing for the lower end of the stirrer rod *F*. The inner tube *E* is four inches long with an outside diameter of 30 mm. and is open at both ends. Its lower end has four small feet which rest on the bottom of the outer vessel. Its upper end has four radial glass projections to hold it in the center of the outer vessel. This arrangement permits efficient circulation of the silicone fluid through the annular space between the inner tube and the outer vessel. Inside the inner tube and about one-third of an inch from its lower end there are four thin glass projections which support the heating coil *R* in place. If the coil is properly shaped it stays in its place without much trouble. In order to make it more secure it may be tied to the feet of the inner tube by small loops of thin steel wire.

The heating coil is made from 22-gage resistance wire. The ends of the coil are silver-soldered to two copper wires which then pass up through two holes in the ceramic tubing *D*. At the upper end of the ceramic tubing these copper wires are connected to an electric cord *A* from a variable transformer, to regulate the rate of heating.

[1] Issued as National Research Council No. 3391.
[2] HERSHBERG, E. B., *Ind. Eng. Chem., Anal. Ed.*, **8**, 312 (1936).
[3] MERRIAM, F. C., *ibid.*, **20**, 1246 (1948).



Melting-point Apparatus

A, Electric cord
B, Holes for melting-point tubes
C, Rubber band
D, Ceramic tubing
E, Inner tube
F, Stirrer rod
G, Clip
H, Aluminum cover
I, Outer vessel
J, Melting-point tube
K, Thermometer guide
L, Anschutz thermometer
M, Aluminum rods
N, Wire gauze
O, Stationary blades
P, Propeller blades
Q, Stirrer blades
R, Heating coil of nichrome wire
S, Bottom well

The stirring mechanism of this apparatus is composed of three different parts. The stirrer blades *Q* are made of thin strips of steel, and each blade is 3 mm. in width. They are sixteen in number and all of them are radially joined to the upper half of a steel collar. This steel collar is about 7 mm. long. It fits loosely on the stirrer rod and can be tightened on the latter at any convenient position, with the help of a set screw placed in the lower half of the collar. The propeller blades *P*, which are also sixteen in number, are similarly radially joined to a steel collar, which can be tightened on the stirrer rod with the help of a set screw. As the function of the propeller blades is to push the silicone fluid up, the angle and the direction of bending of each blade will depend upon the rate of circulation desired and direction of rotation of the electric motor used, respectively. Just above the propeller blades we have the stationary blades *O*, which have the same shape and are same in number as the stirrer blades, but the collar in this case is not tightened onto the stirrer rod. The steel collar is loose enough to permit free rotation of the stirrer rod inside it, but is not so loose as to wobble. These blades are held stationary by the lower ends of the aluminum rods *M*, which come down from the cover *H*. As soon as the heated liquid rises up from the heating coil it is stirred thoroughly and mixed uniformly by the large number of stirrer blades. Soon after that the mixed liquid is lifted up by the propeller blades, which also do some mixing. By now the hot liquid is well mixed and is on its way up toward the region of the thermometer bulb and the bottom end of the melting-point tube *J* containing the sample of substance under investigation. But the whole column has also acquired a good deal of circular motion. The stationary blades help to break this circular motion and after that point a uniformly heated and thoroughly mixed column of liquid should be rising up. It can be expected that at any cross section of this liquid column the temperature will be uniform. This rising column of liquid inside the inner tube is surrounded on the outside by a cylindrical layer of liquid which may be at a slightly lower temperature and is on its way down to circulation around the heating coil once again. In this way the exposure of the liquid column around the thermometer bulb to larger temperature differences is eliminated. The circular piece of wire gauze *N*, which may or may not be soldered to the stationary blades, serves as a guard against any damage to the thermometer or to the melting-point tube in case one of them accidentally slips down from its rubber band. The holes in the wire gauze should be small enough to hold the melting-point tube but large enough to permit a free flow of the circulating liquid. The pieces of the stirring mechanism and the wire gauze should be made of steel or any other white metal or alloy. If made of brass they reflect yellow light all throughout the body of the oil, which might interfere with the observation of the melting point.

The lower ends of the aluminum rods pass through two small cuts in the wire gauze and come down enough to hold the stationary blades. These rods could be made of any suitable white metal or alloy. Slightly above the thermometer bulb there is a thermometer guide *K*, the two ends of which are soldered to the aluminum rods. It can be made of thin wire or a narrow, thin piece of metal. It has a loop for the thermometer, two small loops on each side of the thermometer loop for melting-point tubes, and one loop behind the thermometer loop for the stirrer rod. The guide should be thin so that it does not interfere either with observation or with the flow of the liquid.

The aluminum cover *H* has a thin, short lip inside the edge of the outer vessel in order to hold itself in place without wobbling. This also helps to drain any silicone oil, which is usually wiped off the thermometer when it is taken out, back into the vessel. In the center of the cover there is a hole for the stirrer rod. The hole for the thermometer is in front and has two small holes *B* on each side for melting-point tubes. On one side there is a hole for the ceramic tubing, which is placed on the side so that it does not interfere with the observation of the melting point. The cover is held sufficiently tight with the help of two clips *G* that it does not wobble or rotate. The stirrer rod is a 3-mm. glass or metal rod. Its lower end is smoothly rounded so that it rotates on the glass surface of the bottom well without friction. Its upper end is connected to a light stirring motor with the help of a rubber sleeve *C*. Its length can be determined by the stem length and the convenience of replacement of the thermometer. There should be enough space between the upper end of the inner tube and the cover to accommodate for the considerable expansion of the silicone oil. The apparatus can be properly lighted with an electric light, and a reading lens can be conveniently used if desired. The whole assembly is compact and handy for use by organic chemists.

AIDS TO SEMIMICRO QUALITATIVE ANALYSIS

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FOR the past six years qualitative analysis has been taught to science students at second-year level[2] in this department solely by semimicro methods. More recently, no students taking chemistry courses have carried out any macro inorganic qualitative analysis. Besides resulting in a great saving in expenditure, the small-scale methods have proved satisfactory both for the illustration of the chemistry of inorganic ions and for actual qualitative analysis. Students, once their preliminary and brief unfamiliarity with the manipulation has passed, find the techniques interesting and elegant. The methods described in the textbook[3] recommended to the students have been very considerably modified as a result of teaching experience, and the students now use the textbook in conjunction with a comprehensive set of additional notes. They are, of course, also expected to correlate their practical experience with the theoretical background and with experimental details contained in standard textbooks on qualitative analysis.

One of the drawbacks during the early days of the teaching was the lack of essential apparatus suitable for purely student use. Such pieces as hanging-drop cells and gas absorption apparatus, as described in the literature, were easily breakable and costly; further, the time a student doing 50–60 hours of practical inorganic chemistry per year could devote solely to construction of apparatus was very limited. Over a period of six years' teaching, aided by constructive student suggestions and criticisms, we have evolved several simple and efficient pieces of apparatus suitable for relatively rough wear and lending themselves to easy handling. In passing, it may be mentioned that various difficulties in converting macro inorganic

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[2] That is, after a first-session course which previously comprised macro qualitative analysis, titrimetric analysis, organic qualitative and preparative work, and physicochemical experiments. The macro qualitative analysis has now been replaced by inorganic gravimetric analysis.

[3] BELCHER, R., AND C. L. WILSON, "Qualitative Inorganic Microanalysis," Longmans, Green and Co., Inc., New York, 1946.

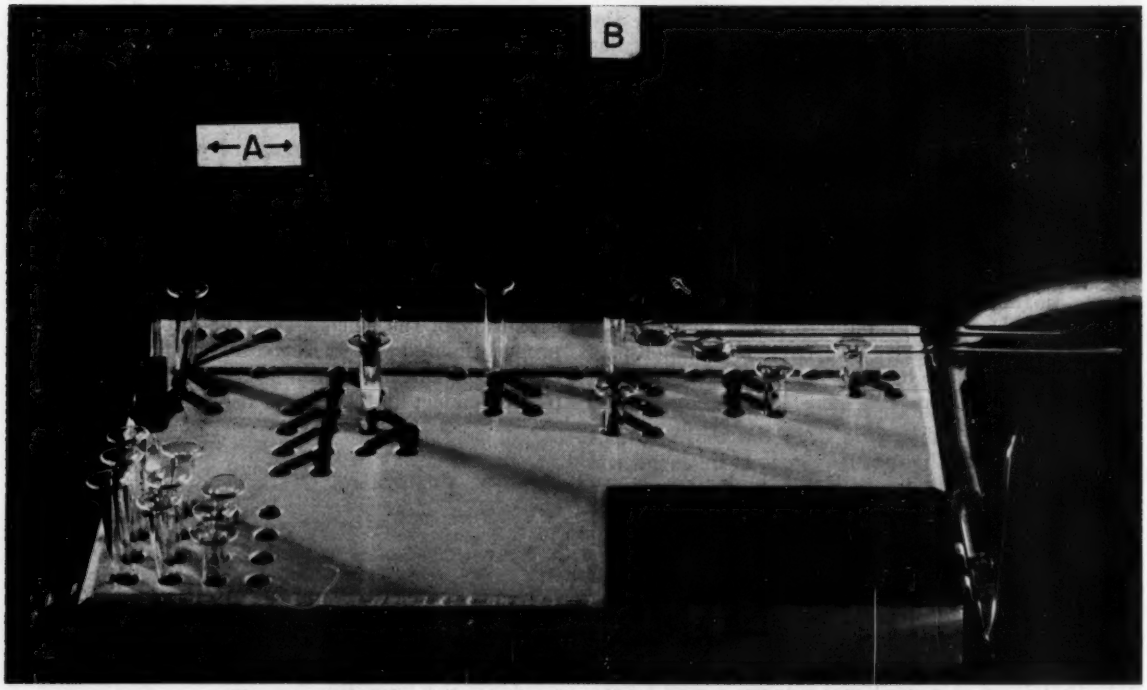

Figure 1. Analysis Board, Type 1, Complete with Apparatus for Which It Is Designed

analysis schemes to semimicro scale have been overcome, and the scheme adopted at present for our course seems quite reliable and easy to carry out.

The manipulative technique and the analysis scheme are constantly under revision and new ideas are continually being tried; it is felt that some of the improvements might well be brought before the notice of other schools of chemistry who are already using semimicro methods or who may wish to adopt such elegant techniques for teaching qualitative analysis.

ANALYSIS BOARDS AND FRAMES

Illustrated in Figures 1 and 2 are two drilled and decorated boards of different design which are useful in the course of an analysis for setting down cones and "spoons," and for carrying, by means of clips, apparatus such as the microburner. Board 1 is constructed for a very rigid scheme of cation separations; the key to this is shown in Figure 3, and does not allow for separation of "less familiar" elements or systematic separation of anions. This is no great drawback, as in this school of chemistry second-year students are not expected to make a rigid identification of the less familiar ions (although they are expected to recognize their presence and to avoid confusing them with the ions usually present in elementary schemes); and an anion group separation is not employed. As a consequence, this type of board is extremely simple and easy to work with; cones containing vital solutions or precipitates are not readily mislaid and all commonly used apparatus is immediately at hand.

Board 2 is less rigid in design and is suitable for any class of separation. It retains the advantages of Board 1, but the student is not tied to a set scheme.

The drilling of these boards, made of rosewood or some similar hardwood, is a wearisome task unless only one or two are required or unless a special jig is made for them. Therefore, wire frames, illustrated in Figure 4, have been issued to the students instead. These are made of stout galvanized iron wire mesh (3 holes per inch) strengthened with a perimeter of $^1/_{16}$-in. diameter wire. The size is 7 × 10.5 in., with 1-in. legs, and therefore they contain approximately 600 useful squares for cones; holes may be expanded, by cutting or bending the wire, to accommodate "spoons." Students are expected to apply one coat of a good lacquer paint, and then may paint on any analysis design they may choose after they have been shown one or two specimen boards and frames. Students are encouraged to work out their own designs, which carries an advantage in that it familiarizes them with the scheme. The completed frame carries a wired-on microburner, and the key to the design is affixed across the base.

HANGING-DROP CELL AND HYDROGEN SULFIDE ABSORPTION APPARATUS

Instead of using separate, specialized apparatus, an attempt has been made to modify existing, commonly used pieces such as cones. The hanging-drop cell, shown in Figures 1 and 5(*A*), is simply constructed from a cone, a platinum wire carried in a glass rod, and a piece of rubber tubing. The manipulation is simple: the reagents are mixed in the cone and a small loose plug of cotton wool is inserted; the rubber sleeve, moistened with the minimum of distilled water, is forced on, and the platinum loop containing the test reagent carefully introduced into the cone so that it can be seen. If necessary, the cone is warmed in a water bath and the wire pushed in still further. The cell may be used with test papers, these being attached to the wire, or placed in a tapered open tube (also shown in Figures 1 and 5(*A*)) that is similar to the tube used for introduction of hydrogen sulfide.

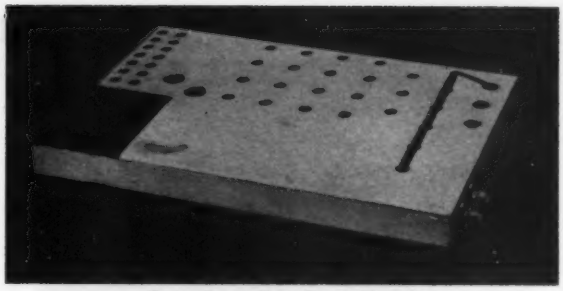

Figure 2. Analysis Board, Type 2

Figures 1 and 5(*B*) show the apparatus for introduction of hydrogen sulfide (or any gas produced in a generator, but not from a cylinder) under pressure. It is advisable to use a cone of relatively flat bottom, and to flick the cone with the finger during gas passage. The inlet tube is of relatively narrow taper, and to prevent splintering it is fire-polished at the ends and cushioned against the cone with a small piece of cycle valve rubber. Operation is again simple and little of the unpleasant gas is allowed to escape. The solution under test is pláced in the cone and the rubber sleeve and cushioned inlet tube are placed in position. The latter is connected to the hydrogen sulfide generator via a purification train; the tap of the generator is opened, and if there are no leaks the inlet tube of the apparatus is mo-

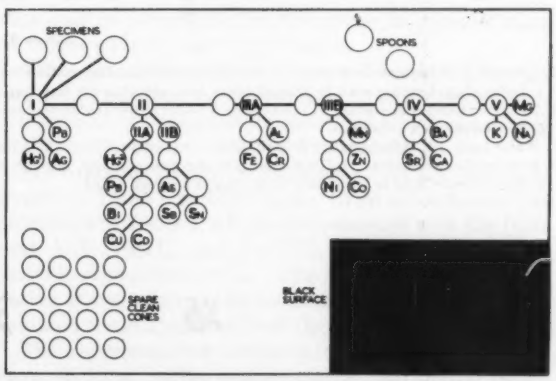

Figure 3. Key to Layout of Analysis Board, Type 1

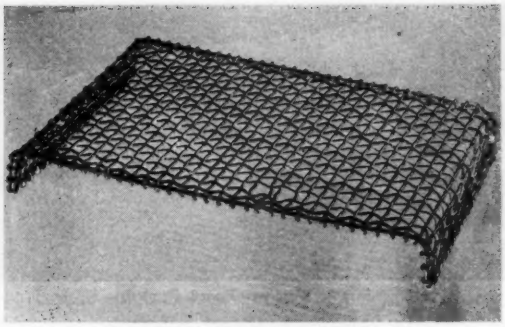

Figure 4. Analysis Frame, Unpainted

mentarily withdrawn so that the air is blown out of the delivery tubing and cone. The inlet tube is replaced, and with occasional flicking and placing of the cone in a hot water bath, a pressure of gas is maintained for the prescribed time (3 to 5 minutes for the copper/tin group, 1 minute only for the zinc group). Besides giving a more efficient precipitation according to physicochemical laws, the quality of the precipitate is good in that it is more granular and readily centrifuged and not as slimy as it is when produced by the less desirable process of bubbling in the gas.

WATER BATHS

The difficulty of using a wire framework, from which water retreats on evaporation, for supports for cones during digestion is overcome by using a cork float. A large cork is cut in a direction parallel to its flat faces and the half-cork is bored, say, with four holes which fit the cones closely and support them by the lips. The float is fitted with a small wire handle and is shown diagrammatically in Figure 5(*C*).

MISCELLANEOUS HINTS

(*a*) Microscope slides cut in half lengthwise are more suitable and less easily broken by heat than full 3 × 1-in. slides.

(*b*) When it is necessary to boil a liquid in a cone, bumping and loss of the solution by spurting may be prevented by placing a finger firmly over the top and keeping it there until the liquid has cooled below its boiling point.

(*c*) During evaporations in "spoons," in order to prevent bumping and to promote rapid evaporation it is convenient to blow a jet of air onto the surface of the liquid through a drawn-out tube (see Figure 5(*D*)). Provided that the jet does not dip under the surface of the liquid, minute bubbles are introduced into the solution and these act as nuclei for uniform boiling.

The pieces of apparatus described above for some of the cardinal manipulations in semimicro analytical procedures are robust, easily manipulated, and readily assembled. Further, they have stood the acid test of student use.

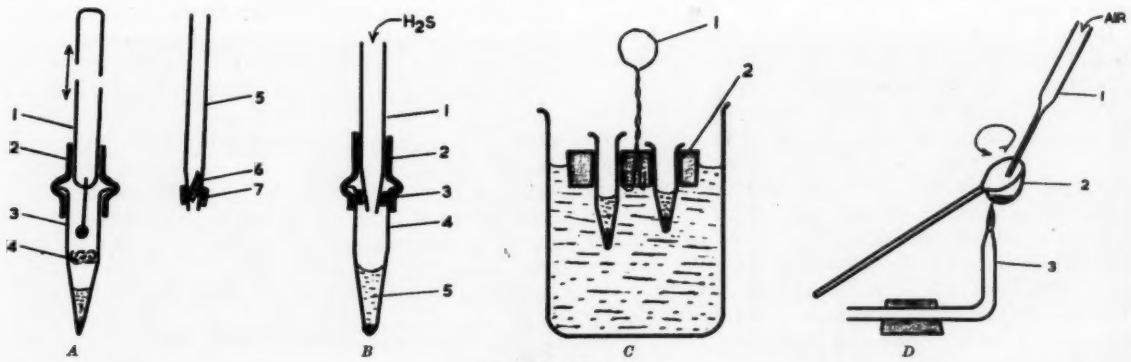

Figure 5. Apparatus

A, Two types of hanging-drop cells. 1, Holder for platinum loop containing test solution. 2, Outer sleeve of rubber tubing. 3, Cone (2–3 ml. capacity). 4, Loose plug of cotton wool to prevent spray from reaching the test reagent. 5, Tapered tubing. 6, Test paper. 7, Cushion of thin-wall tubing.

B, Hydrogen sulfide apparatus. 1, Inlet tube. 2, Outer sleeve of thin-wall rubber tubing. 3, Cushion of thin-wall rubber tubing. 4, Cone (2–3 ml. capacity). 5, Heated solution under test.

C, Water bath. 1, Wire handle. 2, Half-cork.

D, Evaporation procedure. 1, Glass tubing, drawn to a capillary, through which air is blown. 2, "Spoon," continuously rotated above microburner flame. 3, Microburner, held in large cork, or in clips of analysis board.

REGULARITIES AMONG THE REPRESENTATIVE ELEMENTS: THE "PAIRED ELECTRON RULE"

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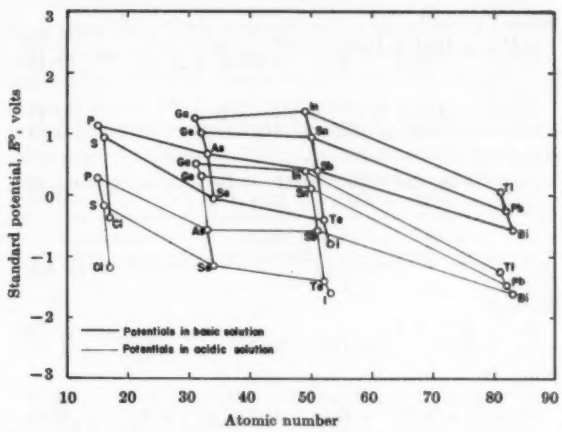
EVEN though most recent textbooks for use in the introductory course mention electron subshells (s, p, d, f), usually in an early chapter on atomic structure, they neglect to correlate these structural features with valences or oxidation states in later chapters devoted to discussion of the chemistry of the several elements. And so the student has to learn *as unrelated facts* that the valences of tin and lead are commonly two and four, that +3 and +5 are oxidation states characteristic of Group V-A elements,[1] that +4 and +6 are oxidation states characteristic of Group VI-A elements, and that +5 and +7 are oxidation states characteristic of Group VII-A elements, except that bromine does not form perbromates.

It is doubtful that any real understanding of valence comes from meeting such facts in isolation. But if the facts are correlated in terms of electron subshells, not only do they become reasonable and logical but also there results an appreciation of atomic structure as something more than mere theory. The principles are shown to have utility and a relationship to actuality.

Table 1 presents a number of redox couples and their standard potentials taken from W. M. Latimer's "Oxidation Potentials" (*1*), or calculated with the aid of thermodynamic data therein, and rounded off, in some cases, to the nearest 0.01 volt. Parentheses indicate potentials estimated from known chemical behavior. The potentials are plotted in the figure against the atomic number of the element being oxidized. The more nearly horizontal lines in the figure connect elements in the same group of the periodic table; the more nearly vertical lines connect elements in the same period. Heavy lines are for potentials in basic solution; light lines are for potentials in acidic solution.

Each potential is for oxidation of a representative element to its highest oxidation number, G (equal to its periodic table group number), from an oxidation number, $G-2$, in pure water, that is, in the absence of complexing agents other than water. The regularity apparent from the figure is illustrative of the well known decreasing stability of the highest oxidation state of the elements in the A subgroups (*2*), and increasing oxidizing power of the higher oxides and their corresponding acids (*1*), with increasing atomic weight. One need only recall the oxidizing power of lead dioxide which makes the storage battery so effective, the ability of



Regularity in Oxidation Potentials of the Couples $E^{G-2} - E^{G} + 2e$ Among Representative Elements. (G = periodic table Group number)

bismuthate to oxidize manganous ion to permanganate, and the utility of arsenate compared with that of phosphate as an oxidimetric reagent, to make this clear.

The regularity apparent in the figure is partly a result of choice of half-reactions. That is, in compiling data for Table 1, an attempt was made to make all the couples pertaining to the elements in any one group, and in either acidic or basic solution, perfectly analogous with respect to the physical state and degree of hydration or ionization of the substances involved in the half-reactions. This was not always possible because of lack of necessary thermodynamic data. The data in Table 2 illustrate the amount of variation in standard potential that may be introduced by differences in physical state or degree of hydration or ionization.

While the amount of variation so introduced may be large, it is felt that valid conclusions can be drawn by a comparison of potentials for reactions which are closely similar with respect to the factors mentioned.

For example, by an "extrapolation" of the data presented in Table 1 and the figure, there are obtained the potentials in Table 3, which are consistent with the facts that Al(I), Si(II), and Br(VII) have never been prepared in aqueous solution. The instability of these oxidation states emerges as a natural consequence of the common trend apparent from the figure.

The representative elements of Groups III to VII have, in their lowest energy states, two paired (spins opposed) s electrons and one or more p electrons in the outer shell, the total number of outer shell electrons

[1] There is disagreement among authors in the usage of "A" and "B" to distinguish subgroups in the periodic table. In this article, as will be clear from the text, "A" is used for representative elements, "B" for transition elements.

TABLE 1

Standard Potentials for the Oxidation, $E^{G-2} \rightarrow E^{G}$, in Aqueous Solution ($G$ = Periodic Table Group Number)

Period 3 Couple	E°, volt	*Period 4* Couple	E°, volt
Group III			
		$Ga_2O + 2H_2O = Ga_2O_3 + 4H^+ + 4e$	(0.5)[a]
		$Ga_2O + 4OH^- + H_2O = 2Ga(OH)_3 + 4e$	(1.25)
Group IV			
		$Ge^{++} + 2H_2O = GeO_2 + 4H^+ + 2e$	(0.3)
		$GeO + 4OH^- = GeO_3^{--} + 2H_2O + 2e$	1.01
Group V			
$H_3PO_3 + H_2O = H_3PO_4 + 2H^+ + 2e$	0.28	$HAsO_2 + 2H_2O = H_3AsO_4 + 2H^+ + 2e$	−0.56
$HPO_3^{--} + 3OH^- = PO_4^{-3} + 2H_2O + 2e$	1.12	$AsO_2^- + 4OH^- = AsO_4^{-3} + 2H_2O + 2e$	0.67
Group VI			
$H_2SO_3 + H_2O = SO_4^{--} + 4H^+ + 2e$	−0.17	$H_2SeO_3 + H_2O = SeO_4^{--} + 4H^+ + 2e$	−1.15
$SO_3^{--} + 2OH^- = SO_4^{--} + H_2O + 2e$	0.93	$SeO_3^{--} + 2OH^- = SeO_4^{--} + H_2O + 2e$	−0.05
Group VII			
$ClO_3^- + H_2O = ClO_4^- + 2H^+ + 2e$	−1.19		
$ClO_3^- + 2OH^- = ClO_4^- + H_2O + 2e$	−0.36		

Period 5 Couple	E°, volt	*Period 6* Couple	E°, volt
Group III			
$In^+ = In^{+3} + 2e$	(0.39)	$Tl^+ = Tl^{+3} + 2e$	−1.25
$In^+ + 3OH^- = In(OH)_3 + 2e$	(1.37)	$TlOH + 2OH^- = Tl(OH)_3 + 2e$	0.05
Group IV			
$Sn^{++} + 2H_2O = SnO_2 + 4H^+ + 2e$	0.10	$Pb^{++} + 2H_2O = PbO_2 + 4H^+ + 2e$	−1.46
$HSnO_2^- + 3OH^- + H_2O = Sn(OH)_6^{--} + 2e$	0.93	$PbO + 2OH^- = PbO_2 + H_2O + 2e$	−0.25
Group V			
$2SbO^+ + 3H_2O = Sb_2O_5 + 6H^+ + 4e$	−0.58	$2BiO^+ + 3H_2O = Bi_2O_5 + 6H^+ + 4e$	(−1.6)
$SbO^+ + 5OH^- = H_3SbO_6^{-4} + H_2O + 2e$	(0.40)	$Bi_2O_3 + 2OH^- = Bi_2O_4 + H_2O + 2e$	−0.56
Group VI			
$H_2TeO_3 + H_2O = TeO_4^{--} + 4H^+ + 2e$	(−1.4)		
$TeO_3^{--} + 2OH^- = TeO_4^{--} + H_2O + 2e$	(−0.4)		
Group VII			
$IO_3^- + H_2O = IO_4^- + 2H^+ + 2e$	(−1.6)		
$IO_3^- + 2OH^- = IO_4^- + H_2O + 2e$	(−0.8)		

[a] Parentheses indicate approximate potentials estimated from known chemical behavior (*1*).

being equal to the group number, G. Presumably all the outer shell electrons are involved when one of these elements forms a compound in which it is in its highest oxidation state, but only the p electrons are involved when the oxidation number is $G-2$. Presumably, also, involvement of an s pair requires unpairing and is accompanied by hybridization of s and p atomic orbitals to form hybridized molecular bond orbitals.

TABLE 2

Effect of Differences in Physical State and Degree of Hydration or Ionization on Standard Oxidation Potential

Couple	E°, volt	ΔE°, volt
$2SbO^+ + 3H_2O = Sb_2O_5 + 6H^+ + 4e$	−0.58	0.10[a]
$2SbO^+ + 2H_2O = Sb_2O_4 + 4H^+ + 2e$	−0.68	
$Sn^{++} + 2H_2O = SnO_2 + 4H^+ + 2e$	0.10	0.25
$Sn^{++} = Sn^{+4} + 2e$	−0.15	
$H_2TeO_3 + H_2O = TeO_4^{--} + 4H^+ + 2e$	(−1.4)[b]	(0.4)
$TeO_2 + 4H_2O = H_4TeO_6 + 2H^+ + 2e$	−1.02	
$Ge^{++} + 6OH^- = GeO_3^{--} + 3H_2O + 2e$	(2.04)	(1.03)
$GeO + 4OH^- = GeO_3^{--} + 2H_2O + 2e$	(1.01)	

[a] From another point of view, the smallness of this difference might be used as justification for calling Sb_2O_4 "antimonyl antimonate."

[b] Parentheses indicate approximate values.

From this point of view, the oxidation potentials of Table 1 are a measure of the lability of an s pair and of the energy released in hybridization. It appears as a general rule that the lability of an s pair decreases with increase in atomic number, both within a period and within a group. The decrease in lability with increase in atomic number within a *period* can be correlated with changes in atomic size, just as the regularities exhibited by the series in Table 4 seem consequent to changes in atomic size. But the trend within each group is in a direction opposite to what would be expected from consideration of atomic radii. For the present, the rule that the lability of an s pair decreases ("inertness" increases) with increase in atomic number within a *group* is presented as a purely empirical one.

A number of illustrations of the rule, other than those involving oxidation potentials, can be given.

(1) Among Group III representative elements, there

is a progressive increase in stability of the monohalides and decrease in stability of the trihalides with increase in atomic number. For example, although there seems to be about 34 mole per cent of aluminum monochloride in equilibrium with the trichloride in the presence of aluminum at 1127°, the monochloride disproportionates at lower temperatures (*4*). Gallium trichloride is reduced by gallium to the dichloride, which is probably a dimer, but no monochloride seems to be formed up to 175° (*5*). Both indium monochloride and indium trichloride are stable at their boiling points (550° and about 600°, respectively (*6*)). Thallic chloride decomposes to chlorine and, presumably, thallous chloride, above 40° (*3*). The extremes of behavior are summarized by the equations:

$$3AlCl \rightarrow 2Al + AlCl_3$$
$$TlCl_3 \rightarrow TlCl + Cl_2$$

(2) Among Group IV representative elements, there is a progressive increase in stability of the dihalides and decrease in stability of the tetrahalides with increase in atomic number. For example, silicon dichloride seems to be formed by reduction of the tetrachloride with silicon at 1000°, but has been obtained only in an impure form and appears to be unstable above 170° (*7*). Germanium dichloride disproportionates to germanium and the tetrachloride in the temperature range 74.6–450° (*8*). Both stannous and stannic chloride are stable at their boiling points (623° and 114.1°, respectively (*6*)). Lead tetrachloride decomposes explosively at 105°, and is partially converted to lead chloride and chlorine by water (*3*). The extremes of behavior are summarized by the equations:

$$2SiCl_2 \rightarrow Si + SiCl_4$$
$$PbCl_4 \rightarrow PbCl_2 + Cl_2$$

(3) The regular increase in metallicity with atomic number, especially within Groups IV-A and V-A, can be regarded as an illustration of decreasing lability of an *s* pair. Metals are characterized by having few electrons (three or less) in the outer shell of the atom. The increasingly metallic natures of germanium, tin, and lead in Group IV, and antimony and bismuth in Group V can be reconciled with this description if the pair of *s* electrons in the outer shell of each of these atoms be regarded as "buried" with inner shell electrons, the depth of "burying" increasing with atomic number within each group.

(4) The chlorides of elements of Groups IV-A, V-A, and VI-A in which the valence (oxidation number) is $G-2$ become increasingly ionic in nature with increasing atomic number within each group as shown by regularly increasing melting and boiling points and electrical conductivities in the molten state. Although such data have been interpreted as an effect of change in ionic size (*3*), it is significant that this increasing ionic nature is not found among chlorides of the elements of Groups III-A, IV-A, and V-A when the valence number is *G*. Illustrative melting and boiling point data (*6*) are assembled in Table 5. Typical conductivity data are: phosphorus trichloride is nonconducting, whereas bismuth trichloride has a specific conductivity of 0.442 ohm^{-1} at 266°; the specific conductivity of lead tetrachloride is only 8×10^{-7} ohm^{-1} (*9*). Thus it appears that in the chlorides of lower valence number the bonding *p* electrons become increasingly differentiated from the nonbonding *s* electrons as the atomic number increases within each group.

It is noteworthy here that the potentials for the formation from the elements of the states with oxidation

TABLE 3
Potentials by Extrapolation

Couple	E° *(extrap.), volt*
$Al^+ = Al^{+3} + 2e$	1.2
$Al^+ + 3OH^- = Al(OH)_3 + 2e$	2.2
$Si^{++} + 2H_2O = SiO_2 + 4H^+ + 2e$	0.8
$SiO + 4OH^- = SiO_3^{--} + 2H_2O + 2e$	1.6
$BrO_3^- + H_2O = BrO_4^- + 2H^+ + 2e$	−2.0
$BrO_3^- + 2OH^- = BrO_4^- + H_2O + 2e$	−1.2

TABLE 4
Oxidation Potential Regularities Dependent on Atomic Size

Couple	E°, *v.*	*At. rad., Å. (3)*
	Series 1	
$Be = Be^{++} + 2e$	1.85	0.90
$Mg = Mg^{++} + 2e$	2.37	1.36
$Ca = Ca^{++} + 2e$	2.87	1.74
$Sr = Sr^{++} + 2e$	2.89	1.92
$Ba = Ba^{++} + 2e$	2.90	1.98
$Ra = Ra^{++} + 2e$	2.92	..
	Series 2	
$2F^- = F_2 + 2e$	−2.65	0.64
$2Cl^- = Cl_2 + 2e$	−1.36	0.99
$2Br^- = Br_2 + 2e$	−1.07	1.14
$2I^- = I_2 + 2e$	−0.54	1.33

TABLE 5
Physical Properties of Chlorides (°C.)

	m. p.	*b. p.*		*m. p.*	*b. p.*		*m. p.*	*b. p.*
			(*A*) *Chlorides of valence number* $G-2$					
			PCl_3	−91	76	SCl_4	−30	Dec.
			$AsCl_3$	−18	130	$SeCl_4$	305	288(?)
$SnCl_2$	246	623	$SbCl_3$	73	223	$TeCl_4$	224	414
$PbCl_2$	501	950	$BiCl_3$	230	447			
			(*B*) *Chlorides of valence number G*					
BCl_3	−107	13	CCl_4	−23	77			
$AlCl_3$	190	183	$SiCl_4$	−70	58	PCl_5	167	162
$GaCl_3$	78	201	$GeCl_4$	−49	83	$AsCl_5$	−40	..
$InCl_3$	586	600(?)	$SnCl_4$	−33	114	$SbCl_5$	3	140
$TlCl_3$	25	Dec.	$PbCl_4$	−15	Dec.			

TABLE 6
Standard Potentials for the Oxidation $E^0 \rightarrow E^{G-2}$

	E°, volt
(A) Group IV-A (G = 4)	
$Ge = Ge^{++} + 2e$	(0.0)
$Sn = Sn^{++} + 2e$	0.136
$Pb = Pb^{++} + 2e$	0.126
(B) Group V-A (G = 5)	
$2As + 3H_2O = As_2O_5 + 6H^+ + 6e$	−0.234
$2Sb + 3H_2O = Sb_2O_5 + 6H^+ + 6e$	−0.152
$2Bi + 3H_2O = Bi_2O_5 + 6H^+ + 6e$	−0.37
(C) Group VI-A (G = 6)	
$S + 3H_2O = H_2SO_3 + 4H^+ + 4e$	−0.45
$Se + 3H_2O = H_2SeO_3 + 4H^+ + 4e$	−0.740
$Te + 2H_2O = TeO_2 + 4H^+ + 4e$	−0.529
$Po + 2H_2O = PoO_2 + 4H^+ + 4e$	−0.7

number $G-2$ in aqueous solution are very nearly the same for all the representative elements in any one group. Typical data are presented in Table 6 (*1*). These data support the thesis that the increased differentiation between *s* and *p* electrons with increase in atomic number within a group is a resultant of decreasing lability of the paired *s* electrons rather than of increasing lability of *p* electrons. These data also raise doubt as to the validity of ascribing the increase in metallic nature with increase in atomic number in Groups IV-A, V-A, VI-A, to a loosening of the outer shell electrons consequent to the increase in atomic size.

TABLE 7
Group II Potentials

Couple	E°, volt
$Mg = Mg^{++} + 2e$	2.37
$Zn = Zn^{++} + 2e$	0.763
$Cd = Cd^{++} + 2e$	0.403
$Hg = Hg^{++} + 2e$	−0.854

Pairing of *p* electrons begins in the representative elements of Group VI, not before (Hund rule). Thus Group VI-A elements have an *s* pair, a *p* pair, and two unpaired *p* electrons, while Group VII-A elements have an *s* pair, two *p* pairs, and a single unpaired *p* electron, in their outer shells. The rule that the lability of an electron-pair decreases with increasing atomic number within a group applies to *p* electrons as well as to *s* electrons. Data that illustrate application of the rule to *p* electrons follow.

(1) The stability of the +1 oxidation state seems to be greater for iodine than for the other halogens. While I^+ compounds have been studied extensively and the ion appears to be stabilized by solvation in pyridine (*10*), Br^+ and Cl^+ are not so well characterized (*11*).

(2) Among Group VI-A elements, "the +2 state is unstable with respect to decomposition into the element and the +4 state, *except in the case of polonium*" (*1*) (italics by this author). Polonium "appears to exist in water solution as +2 polonium, Po^{++}" (*1*).

(3) The metallicity of polonium (*12*) can be reconciled with its having so many as six outer shell electrons if the four paired electrons be regarded as "buried."

(4) The +1 chlorides of Group VII-A elements and the +2 chlorides of Group VI-A elements become increasingly ionic with increasing atomic number within each group, according to evidence from melting and boiling points and electrical conductivities. The following data illustrate this (*6*).

	m. p., °C.	*b. p.*, °C.
OCl_2	−20	4
SCl_2	−78	59
$TeCl_2$	209	327

The specific conductivity of ICl is 4.64×10^{-3} ohm^{-1} at 40°; that of $TeCl_2$ is 0.0420 ohm^{-1} at 206° (*9*).

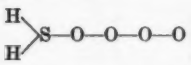
In the light of the foregoing, an empirical generalization, the "paired electron rule," may be stated as follows: *The lability of paired electrons decreases with increase in atomic number within a group of the periodic table, "first-row" (Period 2) elements excepted.* Decreasing *s* pair lability produces trends in oxidation potentials opposite to what would be expected from the concurrent increase in atomic size. The only immediately apparent exception to the rule as it is stated is found among the elements of Group II-A (data of Table 4, Series 1) where the trend is clearly related to atomic size and no decrease in *s* pair lability is discernible. In Group II, the potentials of magnesium and the B subgroup elements (Table 7) form a series analogous to those in Table 1 and plotted in the figure.[2]

The "paired electron rule" may be helpful in teaching atomic structure and valence and the concept of oxidation potential as a measure of chemical stability to first-year students. Much of the information cited in this article is presented in the first-year course, if a typical textbook be taken as an indication of course content; but it is presented as a number of isolated facts which must be remembered with no attempt made to point out the relationships between them. In terms of electron subshells and the "paired electron rule," the "nonexistence" of perbromates and the multivalent nature of tin and lead, for example, can be shown to have a common basis.

No one can deny that the time-honored practice of treating the elements by Groups has pedagogical utility. But it does introduce the danger of overcompartmentalization. The benefits to be gained by a reorganization of the fundamental subject matter into chapters giving "horizontal" coverage should not be overlooked. Conceivable titles for such chapters, in addition to one suggested by this article, are: "Allotropy," "Electronic and structural formulas of covalent compounds," and "Prin-

[2] Authors are in disagreement in the classification of Zn, Cd, and Hg. Despite the link with the representative elements indicated by the data of Table 7, it seems preferable to class Zn, Cd, and Hg with the transition elements or "related metals" (*13*) rather than with the representative elements (*14*), because they form stable complex ions with ammonia and cyanide ion, for example, and because of the catalytic activity of their oxides. These chemical properties are characteristic of the transitional rather than of the representative elements.

ciples of inorganic chemical synthesis." Such a treatment might provide answers to such questions as, "Why is allotropy found chiefly among nonmetallic elements?" and, "Why do the halogens not show allotropy?" which are not answered by the present mode of coverage; it might lead students away from writing such structures as the following for H_2SO_4:



and it might enable them to devise practical syntheses—from the elements, say—for "type" compounds, such as magnesium selenate and gallium tribromide.

Finally, the common practice of placing emphasis on the study of those elements with socio-economic importance and making no mention of the others in the same group can only result in a distorted point of view and is certainly the antithesis of teaching chemistry as a *science*. Can you imagine what kind of a periodic table Mendeleev would have produced if he had limited his treatment to the elements of socio-economic importance in his day? A detailed treatment of highly specialized technological processes of social and economic importance may serve to make the subject interesting for one brand of student. But how many potentially brilliant chemists have become merely good physicists because their naturally orderly minds revolted against the hodgepodge-like fare that was placed before them as the "science" of chemistry? The question, "What is science?" can best be answered by the example of teaching a scientific subject as organized knowledge rather than as technology. A trained paleontologist may be able to reconstruct a reasonably authentic *Eohippus* from a few bones, but it is doubtful that even the most astute first-year college student can perceive the order that pervades the science of chemistry, and that is exemplified by the "paired electron rule," if the subject is taught as a succession of technological processes and reactions that must be memorized.

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A RAPID-ACTING SEMIMICRO VAPOR DENSITY APPARATUS

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THE parts of the apparatus, which is of the Victor Meyer type, are supported on a $^5/_8$-in. hardwood vertical panel, which is screwed to a baseplate of the same material. A side view of the apparatus is given at (*a*) in Figure 1; the appearance of the front of the panel, which is faced with a light-colored plastic ("Formica"), is shown at (*b*).

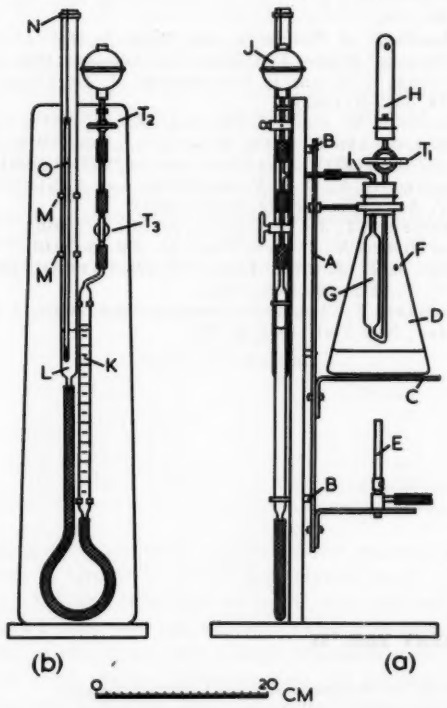

Figure 1. Vapor Density Apparatus

(*a*) side view; (*b*) front view of panel.

A $^3/_{16}$-in. asbestos cement shield *A* covers the back of the panel and is secured by screws passing through distance pieces *BB*. The air space thus formed greatly reduces heat transfer from the hot "vaporizing side" to the cool "measuring side." Flask hotplate *C* is of heavy-gage aluminum sheet and is bolted to the shield. A $2^1/_2$-in. hole cut in the hotplate allows direct access of the flame to the one-liter heavy-duty conical flask *D*. A similar narrower support carries Bunsen burner *E*, which is removed from its base and secured by the base-retaining screw.

Serving as a boiler and steam jacket, the flask has a 3-mm. escape hole *F* blown in the upper part of the wall. If extended runs are envisaged or the use of jacketing substances other than water is required, *F* is replaced by a vertical tube for the attachment of a reflux condenser. Vaporizing chamber *G*, which is surmounted by 3-mm. bore stopcock T_1, is shown on a larger scale at (*a*) in Figure 2. The alternative simpler but not so satisfactory version shown at (*b*) may be improvised from a boiling tube.[2] Either form of vaporizing chamber is supported in the flask by means of a snugly bored cork (for chamber (*a*), the cork is split diametrically) and carries cap *H*. The latter is a 5 × $^5/_8$-in. test tube having a small hole blown in the wall.

The 1.5-mm.-bore exit tube is joined to the 1.5-mm.-bore T-piece *I* (Figure 1), the side arm of which passes through the panel. All such joints are glass-to-glass within tightly fitting sleeves of rubber tubing. Stopcock T_2, which has the same bore as the T-piece, carries a rubber bulb *J*. A similar stopcock T_3 joins the lower arm of the T-piece to measuring pipet *K*. An ordinary 10-ml. graduated pipet with 0.1-ml. subdivisions serves excellently, the mouthpiece being bent as shown. Flow rate must be rapid, so that an ideal pipet is one withdrawn from normal service because of a broken jet. Rubber tubing joins the lower end of the pipet to leveling vessel *L*, which slides vertically in "Terry" spring clips *MM*. Suitably placed but smaller spring clips are used to support the measuring assembly on the face of the panel, while a large clip mounted on a length of brass screw-rod projecting from the shield is used to secure the flask. Stop *N*, cut from rubber tubing, prevents the leveling vessel from being pushed down too far. The stop also anchors the thread supporting thermometer *O*, which dips into colored water contained in the leveling vessel.

The liquid to be examined is weighed into a thin-walled tube that is closed at one end and is about 40 mm. long and 1.5 mm. in bore. The same tube may be used repeatedly, although its weight should be checked from time to time. To prevent chipping when dropped into the apparatus, the open end should be lightly fire-polished. If several tubes are made, they may be dif-

[1] Present address: University of Minnesota, Minneapolis, Minn.

[2] STOCK, J. T., AND C. HEITLER, *School Science Rev.*, **35**, 39 (1953).

Results Obtained with Chloroform (m. w. 119.4)

Run no.	Chamber (a) Observed m. w. (X)	Chamber (a) Deviation (X − x̄)	Chamber (b) Observed m. w. (X)	Chamber (b) Deviation (X − x̄)
1	119.2	−0.6	119.4	−0.8
2	119.4	−0.4	117.4	−2.8
3	119.8	0.0	117.2	−3.0
4	119.8	0.0	120.8	+0.6
5	119.4	−0.4	123.0	+2.8
6	119.7	−0.1	121.3	+1.1
7	120.5	+0.7	122.2	+2.0
8	119.9	+0.1	120.6	+0.4
9	121.9	+2.1	119.4	−0.8
10	119.0	−0.8	122.6	+2.4
11	119.1	−0.7	118.5	−1.7
Av. (x̄)	119.8		120.2	

Standard deviation = $\sqrt{\frac{\Sigma(X - \bar{x})^2}{10}}$ = { Chamber (a), 0.82; Chamber (b), 2.01

ferentiated by sealing a tiny bead of colored glass to the closed end of each.

PROCEDURE

(1) Introduce water (distilled, to prevent scale formation on evaporation) into the flask until the level is about 5 mm. below the bottom of the vaporizing chamber. A wash bottle, the coarse jet of which is inserted through hole *F*, is convenient for this operation. Open stopcocks T_1 and T_3 but close T_2. Heat the flask and adjust the flame height to maintain steady, rather rapid boiling. Move the leveling vessel to bring the meniscus in the pipet to the uppermost graduation.

(2) *Warm* a weighed capillary over a small flame and at once immerse the open end in the sample to be examined. On cooling, a short column of liquid will be drawn in. Using sharp wrist action, shake the column down to the closed end. *Cautiously* reheat until this liquid just boils; at once reimmerse the open end, and thus obtain a longer column. Wipe the exterior of the tube and reweigh.

The amount of sample taken should, on vaporization, displace between 7 and 9 ml. of air, as measured under the prevailing conditions. For a liquid having a molecular weight of 100 the weight required is about 30 milligrams. For very rapid but less precise working the tube is calibrated with mercury, when the weight of sample may be calculated from the column *length*, provided that the density is known.[3]

(3) After the water in the flask has been boiling for at least 10 minutes, close T_1. If no change in the pipet reading has occurred after one minute, remove cap *H* and drop the loaded sample tube, mouth downwards, into the way of T_1. *Slowly* rotate the latter to the open position, when the sample tube will fall into the vaporizing chamber. Close T_1 *as soon as* the tube drops. A downward movement of the meniscus in the pipet should be observed almost at once; then steadily lower the leveling vessel to maintain atmospheric pressure within the apparatus. The rapid movement of the meniscus usually ceases within about 10 seconds after introduction of the sample. Exactly equalize levels, close T_3, and read off the volume of displaced air. Also note the thermometer reading and the barometric height, so that the molecular weight may be calculated in the usual way.[4] Normally, all the readings are completed within one minute of release of the sample tube.

(4) Replace cap *H* and make sure that the bore of the plug of T_1 is in line with the way in the barrel. Open T_2 and squeeze the bulb. The empty sample tube will shoot upwards into the cap and come to rest as shown at (*c*) in Figure 2. While heating, slowly compress fully and release the bulb six times; this will expel the vaporized sample. Remove the cap, take out the sample tube, and make replicate runs as required. About two minutes should be allowed between expulsion of vapor and the introduction of a new sample.

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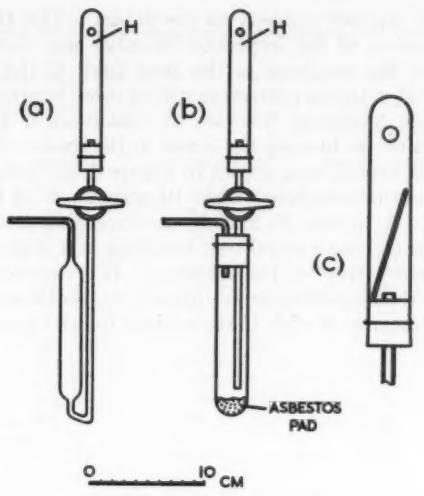

Figure 2. Vaporising Chambers

(a) final form; (b) improvised form; (c) cap, showing sample tube after ejection from apparatus.

RESULTS

First experience of the apparatus was gained using the boiling-tube vaporization chamber, Figure 2(*b*). Results of numerous experiments led to the conclusion that the least satisfactory feature was the delay in establishing temperature equilibrium, or in re-establishing the latter after the introduction of a sample. The improved vaporization chamber has a thin-walled bulb and a greater surface-to-volume ratio than the boiling tube. Further, the tube down which the sample tube is dropped

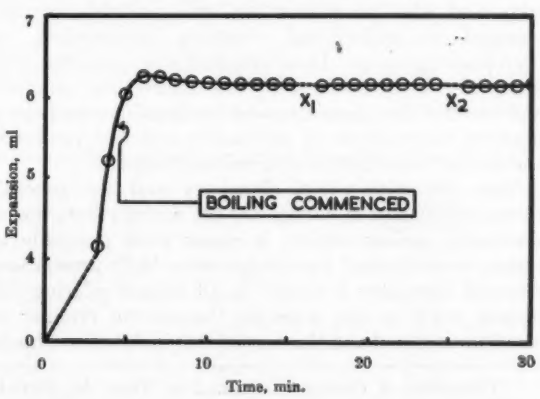

Figure 3. Blank Run with Final Form of Vaporising Chamber

is itself directly exposed to the steam. The thermal performance of the improved chamber was tested by bringing the meniscus to the zero mark of the pipet, closing T_1 with the apparatus still at room temperature, and then observing the rate of expansion of the entrapped air on heating the water in the flask. The result of a typical run, shown in Figure 3, indicates that expansion is complete within 10 minutes after boiling begins. At points X_1 and X_2 the expulsion procedure given in (4) was carried out, resulting in a slight, nonpersistent "creep" of the meniscus. It is therefore concluded that equilibrium is virtually re-established in a time comparable with that required for the manipulation of the various stopcocks, etc. Similar trials with the boiling-tube vaporization chamber showed that noticeable expansion of air continued for several minutes after commencement of boiling, and that slight "creep" persisted even after 30 minutes.

Using pure chloroform as a test substance, the results shown in the table were obtained. Although the runs were performed in groups, the experiments with both forms of apparatus were spread over several days. The results show the decided superiority of the later form of vaporizing chamber.

The authors thank Mr. R. Michell, B.Sc., for experimental assistance.

HOW MANY STUDENTS TAKE GENERAL CHEMISTRY?

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In the work of the Committee on Teaching of College Chemistry of the Division of Chemical Education, it became desirable to have a reliable estimate of the number of students enrolled in general chemistry in the four-year and junior colleges (two-year institutions). Neither the U. S. Office of Education nor the American Chemical Society, nor any other agency, has collected this information nor are they planning to do so. The attention of the Society has been devoted almost wholly to those students who have indicated a major in chemistry and who, on graduating, will probably become engaged as professional chemists, biochemists, or chemical engineers. In an excellent and comprehensive study Dr. B. R. Staneson[1,2] has studied the numbers and trend of the chemistry and biochemistry bachelors, masters, and doctors of philosophy and has predicted the production of future professional chemists.

Since the high-school chemistry and the general-chemistry classes in college are the sources from which chemistry majors evolve, it seems most desirable to obtain some factual knowledge as to their population. General chemistry is taught in all schools offering collegiate work in the sciences; hence the number of teachers required and the cost of this instruction should be of great interest to the Society and chemistry department heads.

To ascertain the number of students enrolled in general chemistry, a sampling procedure was set up to obtain the total number of students enrolled in general chemistry in the fall of 1951 in the 1750 institutions offering this course.

SELECTING SAMPLE OF INSTITUTIONS

A stratified random sample[3] of institutions was obtained from the list of institutions of higher learning wherein the predominant race of students was white, in the 1950 edition of the "College Blue Book." The list was stratified by total enrollment into fifteen strata. An over-all sampling rate of $1/6$ was applied in an attempt to control the size of the sample within each stratum so that not less than 10 per cent nor more than 50 per cent of the institutions listed in each stratum would be selected. It was hoped that the response to the inquiry would result in approximately equal numbers of samples from each stratum, but for various reasons the within-stratum rate varied and the over-all sampling rate was approximately $1/8$. Table 1 gives the stratification and the result of the analysis. The size of each stratum was made to conform with the 1951 census data as reported in Circular No. 36, "Student

[1] "Production of chemists," *Chem. Eng. News*, **30**, 3816–18 (1952).

[2] "Downward trend continues but upturn expected," *ibid.*, **31**, 2376–79 (1953).

[3] Deming, W. E., "Some Theory of Sampling," John Wiley & Son, New York, 1950, pp. 214–15.

TABLE 1
Data for Determining Total Number of General Chemistry Students in the United States in Fall of 1951

Size of institution by total enrollment	*No. of institutions*	*No. of institutions responding to questionnaire*	*Estimated from sample*			*% of total institutional enrollment in general chem.*
			Av. student body size	*Av. enrollment in general chem.*	*Standard deviation*	
<100	207	14	57.4	20.4	14.6	35.6
100– 199	282	30	149.8	25.4	14.6	16.9
200– 299	225	16	232.2	25.6	12.0	11.0
300– 399	152	15	345.5	46.5	23.1	13.5
400– 499	131	13	459.2	63.4	35.9	13.8
500– 599	119	12	260.0	60.4	33.8	10.8
600– 699	89	13	656.6	80.1	51.7	12.2
700– 999	139	12	855.8	87.5	34.6	10.2
1,000–1,499	147	17	1243.9	129.5	76.2	10.4
1,500–2,499	84	13	2017.0	213.8	115.6	10.6
2,500–3,499	50	12	3116.6	427.8	182.0	13.7
3,500–4,499	27	10	4101.2	282.0	228.4	6.9
4,500–6,999	38	14	5786.0	565.0	381.8	9.8
7,000–9,999	28	14	8359.4	595.7	317.7	7.1
>10,000	32	13	14,588.6	739.5	593.0	5.1
Total	1750	218				

Body Size in Institutions of Higher Education for 1951," U. S. Office of Education.

The total number of students in general chemistry for the fall term of 1951 was obtained by a questionnaire sent to these randomly selected institutions.

ANALYSIS OF RESULTS

From the data given in Table 1 and the application of the usual formulas it is estimated that about 186,810 students were enrolled in general chemistry in these 1750 institutions in the fall of 1951.

The standard error of this estimate is 7650, which is 4.1 per cent of the total estimate. Using the two-sigma intervals the estimated total is between the limits of 171,510 and 202,110.

It is interesting to note from Table 1 that as total enrollment increases the percentage of students in general chemistry decreases, probably indicating that the relative drawing power of general chemistry decreases or that the number is being diluted because of the interest and offerings in nonscience fields. It should also be pointed out that the number of students varies considerably in each class although the coefficient of variation is fairly constant. In order to check the representativeness of sample institutions, the total student enrollment in the 1750 institutions was estimated to be 2,002,740, which compares quite favorably to the Office of Education estimate of 2,035,672 (exclusive of nonwhite institutions).

An estimate of the general chemistry enrollment for junior colleges was made in the same way as for all colleges, but using only seven strata (Table 2). The point estimate was 31,479 with a standard error of 1654. Using two-sigma intervals, the enrollment in general chemistry is between 28,171 and 34,787. This estimate represents approximately 21 per cent of the total number of general chemistry students in all institutions in the United States; however, the ratio of students in junior colleges to students in all colleges is approximately 10 per cent.

If the ratio of the number of students reported taking general chemistry in the fall enrollment to the number of first-time students as given by the Office of Education is valid for 1951 and can be used to estimate the numbers of students in general chemistry in the years prior and subsequent to 1951, then the estimated numbers of students in general chemistry are as shown in the fourth column of Table 3 for the years 1947 to

TABLE 2
Data for Determining Total Number of General Chemistry Students in Junior Colleges in the United States in Fall of 1951

Size of institution by total enrollment	*No. of institutions*	*No. of institutions responding to questionnaire*	*Estimated from sample*		*% of total institutional enrollment in general chem.*
			Av. enrollment in general chem.	*Standard deviation*	
<100	128	13	21.4	14.1	35.8
100– 199	152	20	27.7	13.5	19.4
200– 299	76	7	20.3	11.6	12.5
300– 499	77	6	68.2	14.5	17.8
500– 699	39	7	100.3	18.3	17.2
700–2499	50	6	114.5	25.5	10.3
>2500	13	1	565.0[a]	270.0[a]	10.4
Total	535	60			

[a] Estimated from Table 1.

TABLE 3

Number and Relative Percentage of First-time Students in General Chemistry as Determined by Ratio Obtained for Fall, 1951[a]

Year	*Total fall enrollment*	*First-time students*	*Probable no. in general chem.*	*% of students in general chem. to first-time students*	*No. of bachelor's-degree graduates in chem. and biochem.*[b]
1947	2,264,053	569,598	206,295	36.3	7699[c]
1948	2,337,605	549,816	212,947	38.7	9307[c]
1949	2,386,410	538,559	217,412	40.3	10,794[c]
1950	2,226,941	496,835	206,789	41.6	8356[c]
1951	2,050,150	452,741	186,810	41.3	6969[c]
1952	2,079,909	516,602	189,938	36.7	6071[c]
1953	2,181,207	550,693	198,732	36.1	4500[d]

[a] U. S. Office of Education, fall enrollments in higher educational institutions, Negro institutions excepted.
[b] Table 3, *Chem. Eng. News*, **31**, 2377 (1953).
[c] U. S. Office of Education.
[d] Estimate.

1953, inclusive. The percentage of students in general chemistry compared to the fall enrollment of first-time students (freshmen plus transfers) ranges from about 36.1 to 41.6 per cent.

Using the number of bachelor's-degree graduates in chemistry and biochemistry as obtained from the Office of Education and estimated by Stanerson (see the last column, Table 3) the ratio of these graduates to the number of students in general chemistry in the same year ranges from 0.8 per cent (1953) to 2.0 (1949). These are quite questionable values. A better technique is to compare the number of graduates to the number of students in the general chemistry class of four years prior to graduation as set forth in Table 4.

The percentage of graduates of 8356 in 1951 may be high on the base of entering freshmen because of the fact that for a number of years just after the war there were many students returning to college who had taken one or two years of college previously.

TABLE 4

Bachelor's Degree Graduates

	General chem.		*Chem. and biochem.*	*%*
1947	206,295	1951	8356[a]	4.5
1948	212,442	1952	6969[a]	3.3
1949	217,412	1953	6071[a]	2.8
1950	206,789	1954	4500[b]	2.2

[a] U. S. Office of Education.
[b] Estimate.

This drop from 4.5 to about 2.2 per cent is quite significant, considering that the total fall enrollments and first-time students in the past seven years only show a variation of about 15 per cent between the high and low numbers of students.

What are the factors responsible for this drop in bachelor's-degree chemistry graduates—poor quality or lack of inspirational teaching in general chemistry? Are our professors today selling chemistry as effectively as in the past years? Has the decrease in chemistry courses and laboratory sciences in high school reduced the number of college students desiring to major in chemistry?

What are the effects of the draft, World War II, the Korean War, and good wages in the trades and industry? Have we passed the peak and are we now approaching the normal flow of chemistry majors as judged from prewar data?

With about 36 to 42 per cent of the first-time students in general chemistry classes, should not emphasis in these classes be directed toward the cultural aspects of general chemistry and the desirability of today's educated man's knowing something about chemistry, as well as toward prerequisites to other chemistry courses?

In the interest of maintaining a sufficient number of college graduates with majors in chemistry or related sciences to supply the demand of industry and teaching, our Society of 60,000 members should know more about these freshman students and their interests, their high-school preparation, and the influence of the guidance programs in high school and college. We should be aware of the major factors that influence the choices that college students make in selecting their college program. With this information at hand the Society will be in a position to increase interest in science and chemistry and influence the students to study in these fields.

DIVISION OF CHEMICAL EDUCATION

OF THE AMERICAN CHEMICAL SOCIETY

Report of the Treasurer, September 1, 1954

DIVISION ACCOUNT

Receipts:			
Balance on hand 9/1/53		$1,449.01	
Bonds		300.00	
Dues for 1954		907.00	
Dues previous year		61.00	
Dues paid in advance		161.00	
Gifts		6.00	
Subscriptions to the JOURNAL		7.00	
Interest on bonds:		320.00	
$ 7.50 Division account			
312.50 JOURNAL Account			
Total receipts			$3,211.01
Expenditures:			
A. C. S.—Chicago meeting separates	$112.61		
A. C. S.—Receipt cards	37.50		
A. C. S.—Kansas City meeting separates	86.63		
		236.74	
Kansas City Division Dinner Committees	37.50	37.50	
O. M. Smith, Teaching College Chem.	682.18		
L. L. Quill, A.A.A.S. Coop. Com.	31.98		
Columbus Meeting, Nov.	79.40		
Columbus Meeting	25.24		
		818.80	
Chemical Education Publishing Co.			
Subscriptions	7.00		
Reprints Com. Report	33.21		
Reprints Annual Report	8.88		
		49.09	
Transferred to JOURNAL account	312.50	312.50	
Treasurer's Office	241.62	241.62	
Secretary's Office	130.96	130.96	
Total Expenses			$1,827.21
Net balance on hand 9/1/54			1,383.80
Assets distributed as follows:			
Checking account—"Division of Chemical Education, F. B. Dutton, Treasurer"—East Lansing State Bank, East Lansing, Michigan			1,083.80
Three (3) U. S. Savings Bonds, Series G, at $100 ea., Nos. C 825778/80 G. Deposited with R. V. Mellefont, Treas., American Chemical Society, 1155 16th Street, N. W., Washington 6, D. C.			300.00
Total			$1,383.80

JOURNAL ACCOUNT

Balance on hand 9/1/53			
Bonds	$18,000.00		
Cash	4,481.52		
			$22,481.52
Receipts:			
Chemical Education Publishing Co., Easton, Pennsylvania. Net transfer to JOURNAL fund from 1953 profits	2,948.87		
Int. on bank acct.	159.63		
Int. on bonds	450.00		
Total receipts			3,558.50
Expenditures:			
None			
Balance on hand 9/1/54			$26,040.02
Assets distributed as follows:			
Savings account No. 02006, East Lansing Savings and Loan Association, 303 Abbott Road, East Lansing, Michigan			8,040.02
Seven (7) U. S. Savings Bonds, Series G, $1000 ea., Nos. M: 5560890G, 5560861G, 5560855G, 5560856G, 5560857G, 5560858G, 5560859G, deposited with Robert V. Mellefont, Treas., Amer. Chem. Soc., 1155 16th Street, N. W., Washington 6, D. C.			7,000.00
Four (4) U. S. Savings Bonds, Series G, $1000 ea., Nos. M: 5866176G, 5855177G, 5855178G, 5855081G (Deposited with R. V. Mellefont as above)			4,000.00
Four (4) U. S. Savings Bonds, Series G, $1000 ea., Nos. M: 7280064G, 7280066G, 7280067G, 7279936G (Deposited with R. V. Mellefont as above)			4,000.00
Three (3) U. S. Savings Bonds, Series G, $1000 ea., Nos. M: 7438009G, 7438010G, 7437661G (Deposited with R. V. Mellefont as above)			3,000.00
Total			$26,040.02

F. B. DUTTON, *Treasurer*

A RAPID METHOD FOR THE ASSEMBLY OF SEMIDIAGRAMMATIC MOLECULAR MODELS

HANS H. ZINSSER
University of Southern California,
Los Angeles, California

THE transition for students between the conventional letter-type structural formulas and Fisher-Taylor-Hirshfelder type assemblies is at times difficult to make clear. More often is this the case when elementary chemistry has been taught without three-dimensional representations. In many localities the availability of solid models has been a considerable block to the development of such teaching methods.[1, 2]

Originally a bridging technique of the demonstration was derived by straight edge and compass construction of spherical atoms without significant overlap, drawn over staggered letter formulas (see Figure 1). These,

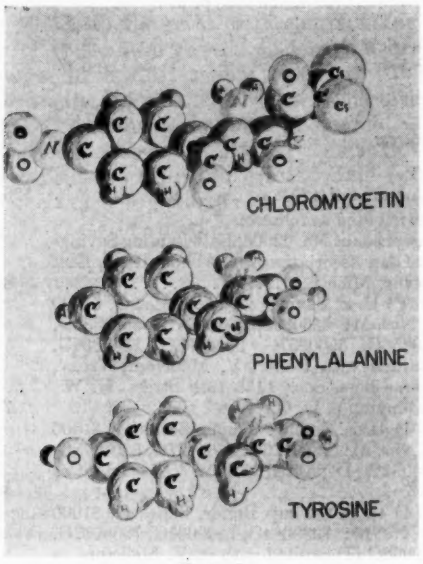

Figure 1

when hand-colored so as to give some shading to the spheres, were readily interpreted by previously untrained audiences and were essentially accurate as far as stereochemistry was concerned, but obviously not adequate for the representation of π orbitals or to demonstrate the contraction effects, which are occasionally important in the understanding of the reaction potentialities of some of the configurations. In order to diminish the time consumed in constructing these and

[1] J. CHEM. EDUC., **28**, 388 (1951).
[2] *Ibid.*, **29**, 249 (1952).

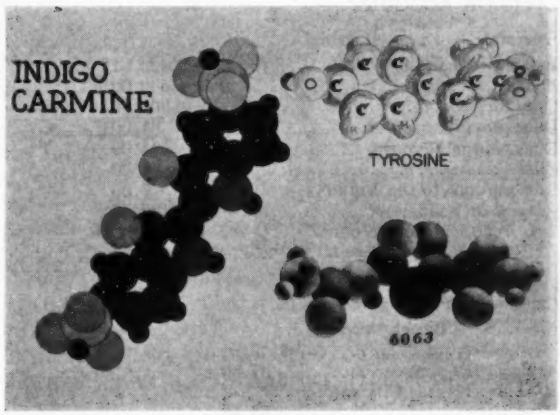

Figure 2

to make trial structures easier to produce, the artifice of using previously colored discs of appropriate size was hit upon and found to be readily available in Scotch-tape type circular labels.[3] With the aid of these labels,

Figure 3

molecules may be assembled at the rate of approximately 1000 atoms per hour, and because they come on sheets in parallel rows, may be readily shaded and colored by air-brush technique by inexperienced operators

[3] These are available in a wide variety of sizes, and special sizes may be made up by the Archer Label Co., 783 Kohler St., Los Angeles 21, Calif.

with only a few hours' practice. The three-dimensional illusion resulting is particularly effective when done in color and used for colored lantern-slide projection. The perspective so derived can be seen in Figure 2. When these are placed on acetate film, their use in the production of animated motion pictures of reaction mechanisms suggests itself. Further use of the air brush to represent π orbitals or areas of high electron density can be carried out with extreme ease, as can be seen in Figure 3. With the wide variety of sizes, either crystallographic projections or true perspective drawings of similar structures can be produced with equal facility and with a minimum of labor (Figure 4). If adequate teaching demands should become apparent, instruction kits for the assembly of some of the commoner structures may be made available on short notice by printing techniques.

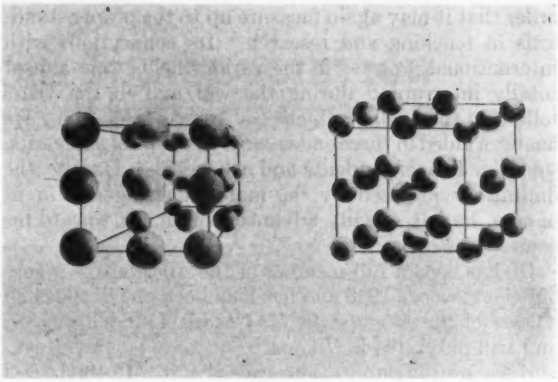

Figure 4

It is to be hoped that this label air-brush technique may serve to facilitate the transition between conventional formulas and true three-dimensional thinking in chemical teaching.

FRIEDRICH WESSELY

FRIEDRICH GALINOVSKY
II Chemisches Universitäts Institut, Vienna, Austria
(Translated by Ralph E. Oesper)

FRIEDRICH WESSELY, born on August 3, 1897, at Kirchberg am Wagram (lower Austria), came from an old jurist family. He served as officer in World War I and was severely wounded toward its close. Following his scientific inclinations, he began his chemical training at Vienna under W. Schlenk and R. Wegscheider. His doctoral work was carried out in the II chemical laboratory of the university under A. Francke. He then went to Berlin, where he continued his training at the Kaiser Wilhelm Institut für Faserstoffchemie under R. O. Herzog. This period, 1923–24, made many decisive impressions on him which became fruitful after his return to his Viennese alma mater. In the meantime E. Späth had become head of the Institut and Wessely served under him from 1927 to 1945 as director of the organic section. In 1929 he habilitated as Privatdozent for organic chemistry and in 1937 was named extraordinary (associate) professor. His researches earned the Wegscheider Prize of the Austrian Academy of Sciences in 1931. A call to head the Institute of Medical Chemistry of the Vienna Medical Faculty was accepted in 1945. He overcame the prevailing difficult circumstances and contributed markedly to the restoration of the department and its laboratory.

Wessely was called back to the II Chemisches Universitäts laboratory in 1948 as successor to the lately deceased Späth. The Institut has borne this name since 1870; it has been headed by many prominent chemists. These include F. Rochleder, one of the founders of phytochemistry; A. Lieben, who directed the Institut for 30 years and who founded a chemical school and contributed many researches of fundamental importance in aliphatic chemistry; Z. H. Skraup, best known for his quinoline synthesis; G. Goldschmiedt, who cleared up the constitution of papaverin; W. Schlenk, whose investigations of free radicals aroused much interest; and finally, E. Späth, who directed the Institut from 1924 to 1946, and who attracted students from all over the world by his researches on alkaloids and other plant materials. Another prominent member of this eminent company was F. Feigl, the renowned master of spot-test analysis and complex-compound chemistry, who worked under Späth until 1938. All these contributed to the high reputation which this laboratory enjoyed before World War II.

Because of the lamentable conditions of the postwar period, Wessely has found it essential since 1948 to carry out a deep-seated reorganization of the Institut in

order that it may again measure up to the proper standards in teaching and research. Its connections with international progress in the various fields were almost totally interrupted during the war and in the years following the conflict which cost Austria so dearly. He has been aided in these endeavors by a corps of assistants, some his former students and colleagues. Despite the limitations imposed by the material stringency in finances, he has already advanced quite far toward his goals.

He has been a full member of the Austrian Academy of Sciences since 1948 and has long been one of the chief editors of the *Monatshefte für Chemie*, founded in 1880 and still published in Vienna.

His scientific publications exceed 120. In them, and corresponding to his many-sided interests, he has not limited himself to a narrow special field of organic chemistry; the topics cover a fairly heterogeneous lot of problems. The earliest studies, which were started while he was at Berlin, dealt with the anhydrides of amino-N-carboxylic acids and their transformation products. Even then he pointed out their significance for peptide syntheses and other questions of protein chemistry, a prediction that has recently been confirmed by the newly awakened interest in these compounds and by further studies in his own laboratory. Other paths for arriving at the constitution of peptides have been opened recently.

Then came his important studies of the constitution of natural vegetable products: glycosides of the coumarin and flavone series, furocoumarins, isoflavones, isoflavanes, bitter principles such as columbin, etc. New kinds of dyestuffs of the type of a phenanthrofuran either were elucidated completely from the structural standpoint or substantial progress has been made along these lines. Collaborating with Späth, Wessely achieved important advances with respect to the clarification of the constitution of podophyllotoxin, which attained considerable importance in cancer therapy.

One series of researches dealt with synthetic estrogens of the stilbestrol type. Pertinent preparative problems were studied, and the steric relations were also investigated in these physiologically interesting classes of compounds. Other studies dealt with such diverse subjects as the separation, identification, and reactions of phenols, as well as their behavior on carboxylation or under the action of lead tetraacetate, and the behavior of the resulting quinols; the polarographic behavior of stilbenes; and dehydrogenations, etc.

It has often been said that the investigational activities and research programs in the European universities are determined almost entirely by the head of the Institut, whose interests and opinions become dominant in the particular local picture. This characterization is not entirely true of the II Chemisches Laboratorium of the University of Vienna. The universal open-mindedness and the wide scientific horizon of its director assure the younger men the opportunity of carrying on their own projects as soon as they have demonstrated their ability. This is why the average output of the past few years has been about 50 papers annually, and many of these bear the names of the younger chemists as sole or principal authors. These papers have dealt, among other things, with paper chromatography and paper electrophoresis or the further development of these techniques. Likewise, inorganic and organic microanalysis is intensively cultivated in this laboratory, following the tradition of the Austrian schools and preserving the tradition of Emich and Pregl. Also traditionally, some of the researches, in addition to studies of peptides and albuminous substances, are concerned with low-molecular natural products, especially alkaloids. Wessely is ready to try out and use the newest methods whenever they are feasible. His lectures include the most recent findings, adding to their stimulus. But like his examinations, these lectures are often quite difficult.

As well as being a versatile scientist, the man himself has a wide spread of interest. He has a horror of dry intellectualism, and this manifests itself in his love of music; he is an excellent performer. This vigor of feeling, which quickly brings his colleagues and collaborators into his circle of friends, has resulted in the creation of a specific "Instituts" atmosphere. His independent attitude toward things and happenings of the day is precisely what makes the scientist Wessely a real teacher of young people. It is not to be expected that a personality so differentiated as Wessely will concur in the usual solutions and slogans of the day.

Although the II Chemisches Universitäts Laboratorium in Vienna has made a nice recovery since the debacle of 1945, it is still faced with the urgent problem of the opportunities open to its well-trained and competent graduates. Still more pressing, because it is crucial for the scientific life of the Institut, is the question as to how capable men can be kept in the school in face of the enticements offered by industry and research foundations, particularly in foreign countries. The fervent hope is that the national authorities and industries, and also those outside Austria, will do their share in the solution of this problem by making research stipends available. The preservation of a high cultural level in Austria is eminently important, and not solely to this country. The United States government and/or its industries might well institute a kind of Marshall Plan by setting up research fellowships in the Austrian schools of higher learning. The returns from an investment of this type could indeed exceed all expectations.

Report of the

NEW ENGLAND ASSOCIATION OF CHEMISTRY TEACHERS

M. S. Tilley

SIXTEENTH SUMMER CONFERENCE

THE Sixteenth Summer Conference of the New England Association of Chemistry Teachers was held August 16–21, 1954, at the University of Massachusetts, Amherst, Massachusetts, with Edward L. Haenisch and Robert D. Eddy as co-chairmen. There were 175 registrants, of whom 56 came from outside the boundaries of New England. States represented included Delaware, Indiana, Maryland, Nebraska, New Jersey, New York, Ohio, Pennsylvania, South Carolina, Virginia, and Wisconsin, as well as the District of Columbia. There were also three registrants from Canada.

The following have attended all 16 summer conferences:

Mr. and Mrs. Grover C. Greenwood, Bridgeport, Connecticut (retired).

Mr. and Mrs. S. Walter Hoyt, Belmont, Massachusetts (retired).

Mr. Alfred R. Lincoln, Hampden College of Pharmacy, Willimansett, Massachusetts.

Miss Evelyn L. Murdock, Stonington High School, Stonington, Connecticut.

Miss Elsie Scott, Northfield School for Girls, Northfield, Massachusetts.

Dr. John R. Suydam, Boston, Massachusetts (retired).

Featured speakers on topics of professional interest were: Arnold J. Currier, "Trends in Chemical Education"; A. B. Hersberger, "Petrochemicals"; Ralph E. Keirstead, "Water, a Basic Natural Resource"; Raymond E. Kirk, "Modern Concepts in Inorganic Chemistry"; William P. Ratchford, "New Wealth from Agri-

culture"; B. R. Stanerson, "The Educational Program of the American Chemical Society"; and Richard Stein, "The Application of Light Scattering Techniques to Chemical Research." For the family audience, John Cavelti described, with magnificent color slides, his "Hawaiian Impressions."

Discussion Groups. Beginning on Tuesday morning, and lasting throughout the week, the membership was broken up into smaller groups for the purpose of carrying out a serious discussion. Each member helped to select the topics to be considered and then had a chance to choose the topic in the discussion of which he particularly wished to participate. Once his selection was made, he was expected to remain with his choice. The topics finally selected and reported on at the last session (in 17 pages of typewritten, mimeographed material) included: "Revision of the NEACT Syllabus";[1] "Admission to College with Advanced Standing in Chemistry"; "Teaching the Quantitative Aspects of Chemistry in the First Course"; "How to Teach Various Concepts (Acid-Base Theories, pH, Hydrolysis, Atomic Theory, etc.)"; "Laboratory Work"; "Physics, Chemistry, and Biology as Separate Subjects Versus a Unified Course in the Physical and Biological Sciences." A coffee break in the middle of each morning discussion period enabled the members in the separate groups to meet together informally, and gave individuals in a particular group a chance to pursue their ideas at greater length.

Exhibits. Exhibits of apparatus, materials, and books were provided by the following organizations: Aloe Scientific Company, Appleton-Century Crofts, Inc., J. T. Baker Chemical Company, The Blakiston Company, Corning Glass Works, E. I. du Pont de Nemours and Company, D. C. Heath and Company, Henry Holt and Company, Inc., Kennecott Copper Corporation, Kinney Manufacturing Company, Mallory-Sharon Titanium Corporation, McGraw-Hill Book Company, Inc., Minneapolis-Honeywell Regulator Company, Reinhold Publishing Corporation, Rem-Cru Titanium, Inc., Technical Operations, Inc., Titanium Metals Corporation of America, Tracerlab, Inc., Watertown Arsenal Laboratory, W. M. Welch Manufacturing Company, and Whitehead Metal Products Company.

Social Program. A carefully organized guests' program, under the direction of Mrs. Walter G. Stewart, kept the wives and families of the members busy throughout the week. Morning trips were taken to historic Deerfield and Mount Sugar Loaf, to a paper processing mill, White and Wyckoff, Inc., where stationery was being printed and boxed, and to a fabric salesroom specializing in mill-ends and remnants. An all-day trip to Old Sturbridge Village was the high point in this supplementary program, and will be long remembered by those who participated. The teen-age guests had their own program of activities under the direction of Guy F. Burrill.

Guests and members together participated in a complete social program under the direction of Angela M. Trovato. In addition to the traditional songfests, theater party, and twisted-whist party of previous years, a new tradition was established with a very successful square dance. It developed that several members of the group had had experience as square dance teachers and callers, and all joined in to make this activity particularly enjoyable. A fabulous picnic of fried chicken, held in the Rhododendron Garden, maintained the very high standards set in this department by previous conferences.

The Sixteenth Conference will be remembered by all for the attractive new facilities and the gracious hospitality provided by the University of Massachusetts and its host, Professor Walter S. Ritchie.

[1] J. CHEM. EDUC., **27**, 46 (1950).

ADJOURNED ANNUAL MEETING, AUGUST 20, 1954

The minutes of the annual meeting of May 15, 1954, were summarized and the list of officers for 1954–55 was read after Marco Scheer, Vice-President, opened the meeting at 2:10 P.M.

The following new members, joining since the May meeting, were welcomed into the Association by the vice-president after the secretary read the names:

Brother Raymund, F.S.C., Head of Science Department, St. Mary's High School, Waltham, Massachusetts.
Charles R. Hurst, Science Teacher, Horace Mann School, New York, New York.
Edmund F. Lankowski, Science Teacher, West Haven High School, West Haven, Connecticut.
Warren Edward Thamarus, Kents Hill School, Kents Hill, Maine.

The following new members joined at the Summer Conference:

Keith D. Crane, Assistant Professor, Otterbein College, Westerville, Ohio.
Anna Jane Harrison, Professor of Chemistry, Mt. Holyoke College, South Hadley, Massachusetts.
Walter William Harvey, Associate Professor, St. Lawrence University, Canton, New York.
Grant W. Smith, Professor of General Chemistry, Pennsylvania State University, State College, Pennsylvania.
Walter B. Spencer, Teacher of Chemistry and Physics, Mount Hermon School, Mount Hermon, Massachusetts.

Treasurer's Report. The treasurer's report, approved by the auditor, S. Walter Hoyt, was accepted. An abridged statement follows:

Balance, August 20, 1953		$1216.34
Receipts, 1953–54		
Dues	$2260.51	
15th Summer Conference surplus	215.75	2476.26
Total		$3692.60
Expenditures, 1953–54		
JOURNAL OF CHEMICAL EDUCATION	$1437.50	
News Letter	228.80	
Endowment Fund	45.68	
Operating Expenses	674.86	$2386.84
Net Balance, August 9, 1954		$1305.76

CARROLL B. GUSTAFSON
Treasurer

Robert D. Eddy, Co-Chairman of the Summer Conference, reported an attendance of 175 at the 16th Summer Conference, of which 100 were members and 75 guests. Of these 119 were from New England.

Vice-President Marco H. Scheer announced that an invitation to hold the 17th Summer Conference at Tufts College, Medford, Massachusetts, from August 15 to 21, 1955, had been accepted. Roland M. Whittaker, Queens College, is to be program chairman and Angela M. Trovato, Robinson Seminary, Exeter, New Hampshire, will serve as chairman of arrangements.

With appropriate comments, Dorothy W. Gifford, Immediate Past President, presented honorary membership certificates to Laurence S. Foster and Mary L. Sherrill. Two other recipients of this award were unable to be present for the ceremony: Grover C. Greenwood and Raymond L. Tobey.

NECROLOGY

JOSEPH SCUDDER CHAMBERLAIN. The necrology committee has been notified rather belatedly of the death, May 13, 1953, of our former president (1928–30) and honorary member (1935), Dr. Joseph Scudder Chamberlain.

Born in Hudson, Ohio, in 1870, he received his B.S. degree from Ohio State College in 1890 and his M.S. degree in 1892. After several years of teaching at Iowa State College (1894–97) and Oberlin (1899–1901), he served as assistant to Dr. Ira Remsen, at The Johns Hopkins University in 1901, receiving his Ph.D. He then was employed as a physiological chemist in the United States Department of Agriculture until 1909.

From 1909 until his retirement in 1940, Dr. Chamberlain served in various grades in the chemistry department of Massachusetts State College, becoming the head of the department in 1928. He was the author of a textbook, published in 1921, entitled "Organic Agricultural Chemistry, an Elementary Biochemistry for Use in Colleges." He joined the New England Association of Chemistry Teachers in 1922 and the American Chemical Society in 1899, serving the Connecticut Valley Section as chairman.

After his retirement in 1940, Dr. Chamberlain lived in Baltimore, Maryland. Older members of the Association will remember the loyal service rendered the New England Association of Chemistry Teachers by Dr. Chamberlain while president; his cordial and friendly manner at the meetings where he presided made all realize that here was an outstanding teacher and gentleman.

S. WALTER HOYT
Chairman, Necrology Committee

MEETINGS SCHEDULED

October 9, 1954, New Hampton School, New Hampton, New Hampshire.
December 4, 1954, Wentworth Institute, Boston, Massachusetts.
February 12, 1955, Mitchell College, New London, Connecticut.
April 23, 1955, St. Joseph College, West Hartford, Connecticut.
May 14, 1955, Framingham State Teachers College, Framingham, Massachusetts (Annual Meeting).

LEALLYN B. CLAPP, *Secretary*

To the Editor:

Here is a problem for a chemist. It is common experience to discover that the erasers with which pencils come equipped often "go bad," that is, they become hard and do not perform their function. The mechanism of what happens I do not fully understand, but suffice it to say that in these parts rubber suffers unduly from atmospheric effects. I believe that automobile tires show similar accumulative effects. Now the problem I wish to propose is this: Some years ago I acquired a stock of brand-new pencils, all equipped with good usable erasers. About half of these were at one time put into a trunk (on the floor of the trunk) wherein resided a heavy concentration of moth repellent (moth balls). The rest of these pencils were in another trunk where no moth stuff was placed. On removing these pencils some years later from their storage in the two trunks I was quite astonished that those which had been in the para-dichlorobenzene atmosphere (I guess it was this) were still in perfect condition, while those in the other trunk were now useless. What is the chemistry of this preservation, apart from the obvious fact that these pencils were not in the usual atmosphere?

JULIUS SUMNER MILLER

EL CAMINO COLLEGE
EL CAMINO COLLEGE, CALIFORNIA

To the Editor:

Recent work by Cunningham, Feay, and Rollier (*J. Am. Chem. Soc.*, **76**, 3361 (1954)), in which they describe the preparation of TbF_4 and their lack of success in an attempt to prepare PrF_4, suggests that an error in the calculations of Perros, Munson, and Naeser (J. CHEM. EDUC., **30**, 402 (1953)) should be called to your attention. The latter make the observation that the differences between the heat of formation of the trichloride and that of the triiodide are constant for several lanthanide and actinide cations. From this they reasonably assume that the difference between the

heats of formation of trichlorides and trifluorides is a constant and estimate the heat of formation of PrF_3. However, they next assume that the difference between the heats of formation of trichlorides and tetrafluorides is constant. This is incorrect since the cations being considered are not the same, but have undergone oxidation to the 4+ state. This means that the energy of oxidation, which differs considerably among the various cations, must be considered. The values given in their paper for the differences between the heats of formation of the trichloride and tetrafluoride of uranium, 230 kg.-cal./mole, and neptunium, 212 kg.-cal./mole, do not agree nearly so well as the constant differences quoted between the various trihalides. However, if one corrects these numbers by subtracting the difference between the heats of formation of the 3+ and 4+ ions in solution, namely 24 and 6 kg.-cal./mole in the respective cases (LATIMER, "Oxidation Potentials," 2nd ed., pp. 302–5) a new constant, 206 kg.-cal./mole, is obtained, the same in both cases. If one combines this constant with the value of the oxidation potential, $E^0 = -2.86$ v., given for the Pr^{+3}–Pr^{+4} couple (LATIMER, p. 294), and makes the rest of the calculations as indicated by Perros, Munson, and Naeser, one obtains the value $\Delta F^0 = -1 \pm 24$ kg.-cal./mole for the reaction

$$PrF_3(c) + {}^1/_2F_2(g) = PrF_4(c)$$

rather than the value $\Delta F^0 = -72$ kg.-cal./mole. The results of Cunningham, Feay, and Rollier are consistent with this calculation.

I would like to thank Dr. Perros for a letter in which he agrees with the above correction in the calculational procedure, and also for pointing out a numerical error in the calculations I had originally made.

DOYLE BRITTON
CALIFORNIA INSTITUTE OF TECHNOLOGY
PASADENA, CALIFORNIA

To the Editor:

While soap bubbles blown by exhaled air are heavy and descend to the floor, those blown by hydrogen gas previously inhaled and stored in the lungs readily float up to the ceiling. When the air in the lungs is only partially displaced by hydrogen gas, bubbles of intermediate buoyancies are obtained. Some remain stationary in mid-air or move about sluggishly in the absence of air currents. When this hydrogen technique is concealed from the audience there is an element of surprise in the experiments.

It is important that the hydrogen used for the experiments should be freed of all poisonous impurities such as arsine and others, if present, by storing the gas over alkaline permanganate solution in aspirator bottles.

ARCOT VISWANATHAN
SYED AZMATULLAH

THE NEW COLLEGE
MADRAS, SOUTH INDIA

HEAT TRANSMISSION

William H. McAdams, **Professor of Chemical Engineering, Massachusetts Institute of Technology. Third edition. McGraw-Hill Book Co., Inc., New York, 1954. xiv + 532 pp. 16 × 24 cm. $8.50.**

SINCE 1932, when the first edition of "Heat Transmission" appeared, each decade has seen a revision and modernization of the book, the second edition in 1942 and now the third edition, each sponsored by the Heat Transmission Committee of the National Research Council. The third edition has expanded to 532 pages, compared to the 459 pages of the second edition. Each edition has been divided into the same three basic parts: heat transmission by conduction, radiation, and convection.

For conductive transfer, the chapters have been renamed Steady Conduction and Transient Conduction to emphasize the markedly different conditions under which heat moves. It is immediately noticed that the illustrations used in the second edition have been increased in size in the new edition and new figures illustrate new methods of solution of unsteady state flow problems. As well as serving as a text, the book also serves as a review article which makes reference to the recent important articles, both in theory and in application.

Chapter 4 on Radiant-heat Transmission is again written by H. C. Hottel, who has enlarged it from the 42 pages of the second edition to 71 pages. New charts have been prepared to give the emissivity of some gases up to 5000°F., or rocket gas temperatures. A new method using determinants permits solution of difficult radiation problems.

For convective transfer, the eight chapters of the old edition

have been increased to eleven by the addition of chapters on Natural Convection, Compact Exchangers; Packed and Fluidized Systems, and High-velocity Flow; Rarified Gases. Much new material appears in all chapters, older references have been deleted, and the more recent and better correlations have been used and cited in the bibliography. References now are given by chapters rather than for the book as a whole. New illustrations and student problems are presented.

The Appendix contains many new data on thermal conductivities and physical properties necessary for heat flow calculations.

The reviewer must rescind his recent generalization that the M. I. T. group should learn to spell. Professor McAdams has spelled *mole* correctly through the book.

The new edition of "McAdams" will be welcomed by both teachers in chemical engineering and practicing engineers. Its fundamental nature makes it a popular text in senior and graduate courses. These same fundamentals are needed by the engineer in industry, who will also find useful the original references and illustrated problems.

KENNETH A. KOBE

UNIVERSITY OF TEXAS
AUSTIN, TEXAS

● AUTOTROPHIC MICRO-ORGANISMS

Edited by *J. L. Peel* and *B. A. Fry*, University of Sheffield, England. Cambridge University Press, London, 1954. viii + 305 pp. Many figs. 16 × 25 cm. $5.

UNITED under this one cover is the more pertinent knowledge to date of autotrophic organisms and their mechanisms. There are approximately 800 references, and included within the scope of autotrophism are photosynthesis, the physiology and biochemistry of chemo- and phototrophic autotrophs, and relationships between autotrophic and heterotrophic microorganisms, *e. g.*, bacteria, green algae, and blue-green algae.

Each of the several chapters has been written by an author who may be considered a leader in his field. The chapters were presented as papers at the Fourth Symposium of the Society for General Microbiology, which was held in London in April, 1954. The publishing date, May 19, 1954, indicates the interest of the editors in distributing in one volume the text of the Symposium.

The bulk of the text is composed of serious papers for the advanced student in microbiology and biochemistry. Various groups or organisms such as *Thiobacillus*, photosynthetic bacteria, nitrifying bacteria, green algae, and blue-green algae have been considered as separate chapters. Also chapters such as Utilization of Organic Compounds by Photosynthetic Bacteria and Problems in the Mass Cultivation of Photo-autotrophic Microorganisms have been included. To complete this interesting reference book the introduction and concluding chapters have been well chosen. The first chapter enters into a discussion of the separation between the autotrophic and heterotrophic mode of life. The following quotation illustrates a basic point in the introduction (presumably the statement applies only to bacteria): "The ability to live autotrophically is, as far as is known at present, more common than the necessity to live autotrophically."

As a fitting conclusion K. R. Butlin and J. R. Postgate have supplied a chapter on the economic importance of autotrophic microorganisms, and thus is reviewed the economic end point of autotrophic metabolism. The concluding remarks of the last chapter require no explanation. "The evolution of industrial civilization on this planet has led to a squandering of the more accessible forms of chemical energy (represented by fuels, sulphur, food and other materials of high chemical potential), most of which have accumulated autotrophically over geological eras of time. Already this phenomenon is being reflected in world shortages of various classes of food and raw materials. As these concentrated reserves of accumulated chemical energy become exhausted, it may well become increasingly necessary to utilize deliberately the chemo- and photo-autotrophic microorganisms for their replenishment."

The book is well written and edited. Its place on the serious microbiologists' bookshelf should be most valuable, as it groups information which has heretofore been widely scattered in the literature.

CARL L. OPPENHEIMER

SCRIPPS INSTITUTION OF OCEANOGRAPHY
LA JOLLA, CALIFORNIA

● PRINCIPLES OF POLYMER CHEMISTRY

Paul J. Flory, Professor of Chemistry, Cornell University. Cornell University Press, Ithaca, New York, 1953. xvi + 672 pp. 146 figs. 16 × 24.5 cm. $8.50.

PROFESSOR FLORY has written the most comprehensive treatment of polymer chemistry yet available. His treatment of the subject is necessarily modern since the quantitative aspects of polymer chemistry have only been developed in recent years and Professor Flory himself has been one of the leading contributors to this field.

The book is divided roughly into two parts. The first part deals with the synthesis of polymers particularly from the point of view of reaction mechanisms and kinetics. The second part deals primarily with the thermodynamical and hydrodynamical properties of polymers. Particular emphasis is placed on solution properties as they are applied to the determination of molecular weights. Each chapter, of which there are fourteen in all, is followed by a bibliography of from 25 to 100 or more journal references.

The first chapter is a historical introduction to the subject. It is pointed out that inaccuracies in the determination of the order of magnitude of the molecular weights of polymers considerably retarded the field. It is surprising that the author does not mention at this point the contributions of Einstein on the theory of light scattering, diffusion, and viscosity of solutions which, if properly applied at the time they were published, would have resolved the problem.

The second chapter is concerned with a classification of polymeric substances. The text is accompanied by very comprehensive tables, a feature that is present throughout the book. In the third chapter the author presents considerable evidence to show that every functional group has equal reactivity at all stages of polymerization. This argument is extremely important in order to apply statistical reasoning to the kinetics of polymerization and of depolymerization. The fourth chapter deals with vinyl polymerization involving free radical mechanisms, and the mechanism of initiation is treated in some detail. The inhibiting role of oxygen is, however, treated in a rather cursory manner. Then follows a chapter (Chapter 5) concerned with the mechanism and kinetics of copolymerization, emulsion polymerization, and ionic polymerization. In Chapters 3 and 4 the author makes frequent reference to diffusion-controlled processes which he later develops in Chapter 5. It would perhaps have been more useful if he had devoted a single chapter to this general problem and developed the subject from elementary considerations. The sixth chapter, dealing with the structure of vinyl polymers, includes some results of X-ray diffraction of fibers which could have been elaborated further with the use of drawings of the structures. A later chapter (Chapter 9), dealing with nonlinear polymers and containing a kinetic treatment of branching and cross-linking, is the most original section of the book.

The second part of the book will probably be less comprehensible to the average graduate student of chemistry. It presupposes some knowledge of the statistical-mechanical theory of solutions. The final chapter (Chapter 14), dealing with the frictional properties of polymers in solution, is the most difficult to treat exactly. Despite the high mathematical level of these

latter chapters, Professor Flory has given a clear and reasonably self-contained treatment of a necessarily complicated subject.

The book is worthy of being called a landmark of chemical literature. It is warmly recommended to beginners as well as specialists who wish to acquire a complete picture of the status of the field of polymer chemistry to date. It might also be useful for supplementary reading in graduate courses in thermodynamics and kinetics and could serve as a textbook for a one-year graduate course in polymer chemistry.

GERALD OSTER

INSTITUTE FOR POLYMER RESEARCH
BROOKLYN, NEW YORK

● AMERICAN CHEMICAL INDUSTRY. VOLUMES I AND V

Williams Haynes. **D. Van Nostrand Company, Inc., New York, 1954. Vol. I, lxvii + 512 pp. Vol. V, li + 622 pp. Many portraits. 17 × 25 cm. $15 per volume; set of six volumes, $76.**

THESE two new volumes in the series on "American Chemical Industry" represent an excellent contribution to the history of chemicals in this country. Volume I, covering the period 1608–1910, describes the background and beginnings of the industry from potashes, naval stores, and indigo in colonial times to the hundreds of chemical products manufactured in 1910. This volume makes fascinating reading as it traces the technical and mercantile developments from by-products of land-clearing operations to big tonnage staples.

Of interest are the Chemical Chronology and Appendixes covering statistics through 1910 and several early descriptions of the process details for making potash, indigo, salt peter, and gunpowder. The extensive bibliography and name index are helpful.

Volume V covers the "Decade of New Products: 1930–1939" and describes the second decade of the two between the wars, during which the chemical revolution was completed. During this period synthetic organic chemistry became firmly entrenched and synthetics such as plastics, fibers, insecticides, etc. became commodities. The technical developments of this period are described in terms of companies, personalities, education, and depression economics.

Appendixes in Volume V include the continued Chemical Chronology, business statistics, and interesting passages from NRA codes, tariff testimony, trade agreements, etc.

The six volumes of this work now completed represent a tremendous and successful effort to explore the history of one facet of American industry.

S. RICKLIN

RICKLIN RESEARCH ASSOCIATES
PROVIDENCE, RHODE ISLAND

● METALLURGY OF THE RARER METALS. NUMBER 1: CHROMIUM. NUMBER 2: ZIRCONIUM

H. M. Finniston, **Head of Metallurgy Division, Atomic Energy Research Establishment, Harwell, England, General Editor, Butterworths Scientific Publications, London, and Academic Press Inc., New York, 1954. 22.5 × 15 cm. Number 1, "Chromium," *A. H. Sully.* xii + 272 pp. 137 figs. 29 tables. $5.50. Number 2, "Zirconium," *G. L. Miller.* xviii + 382 pp. 120 figs. 122 tables. $7.50.**

THESE two volumes are the first of a new series of authoritative treatises on the metallurgy and properties of the rarer elements. Each volume is the product of a specialist on a particular element. Subsequent volumes will cover titanium, molybdenum, platinum and allied metals, manganese, and uranium. Much new knowledge about these metals was accumulated during World War II and a large percentage of the government research and development funds spent on metallurgical projects since the war, both here and abroad, has been directed toward increasing knowledge concerning them. This is because of potential and actual application to new military devices such as rockets, jet propelled aircraft, guided missiles, and especially atomic bombs and nuclear reactors for atomic power plants. Industrial applications of these less familiar metals have also been stimulated by the new knowledge that has resulted.

Even though *chromium* is a familiar element to the layman, the development of pure chromium and of chromium-base alloys has been impeded by their characteristic brittleness. "Chromium in a relatively pure condition may be deformed when hot, and. . .worked into sheet and to rod by conventional metal-working methods. . . . Nearly all investigators who have worked with pure chromium have remarked, however, that it is extremely brittle at room temperature. In this respect its behavior is most interesting and still incompletely understood. . . . It may be sawn, turned, drilled and tapped, yet in tension or in bending it usually fractures with zero ductility. . . . This disadvantage, more than any other, has limited the practical application of chromium and chromium rich alloys" (pages 128–9). This aspect of chromium and the origin of brittleness in other metals have been thoroughly reviewed in the volume on chromium (pages 131–52). The extractive metallurgy of chromium and the physical metallurgy of chromium alloys are presented interestingly and thoroughly. This book should stimulate research interests in this difficult but promising element.

The nuclear, chemical, and mechanical properties of *zirconium* suggest that it is the ideal metal out of which to construct nuclear reactors. Zirconium has a favorably low "cross section" for neutrons, is strong, corrosion resistant, and capable of being formed into sheet, rod, and wire. In an inert atmosphere it can be welded into structural shapes. It always occurs, however, contaminated with a small percentage of hafnium which is extremely difficult to separate. Unfortunately, hafnium is a "poison" in a nuclear reactor. A great deal of effort in recent years has been directed toward the development of an economical, commercial method to effect this separation, yet the most recent reference cited in the review of methods of separating these twin elements is dated 1951; and while there is one reference to a 1949 Harwell report, there are none to U. S. A. E. C. documents. It may be assumed that such documents are so inaccessible in the United Kingdom that in books for the British public it is not warranted to mention them.

Otherwise, the chemistry and metallurgy of zirconium and its compounds and alloys are thoroughly covered and are up to date. While the publicity about zirconium has not been so flamboyant as recent public releases on titanium, the commercial development of ductile zirconium in quantity has been an equally noteworthy achievement.

Both of these volumes are highly recommended.

LAURENCE S. FOSTER

BELMONT, MASSACHUSETTS

● THE SULPHUR DATA BOOK

Compiled by the Technical Staff, Freeport Sulphur Co.; edited by *William N. Tuller.* McGraw-Hill Book Co., Inc., New York, 1954. vii + 143 pp. 16 × 24 cm. $5.

"THE SULPHUR DATA BOOK" is a comprehensive and critical compilation of the physical and chemical properties of sulfur, reaction thermodynamics, and solubility. One chapter is devoted to the analysis of sulfur.

ANNUAL INDEX—JOURNAL OF CHEMICAL EDUCATION

VOLUME 31, 1954

The annual index for Volume 31 of the JOURNAL OF CHEMICAL EDUCATION is divided into several parts. Authors are listed in three sections: authors of articles, books reviewed, and letters to the editor. The subject index includes both subjects and titles to articles; the books reviewed are listed in a separate section. The principal subject entries are distinguished from titles by printing the former in italics. Portraits, including medals, are indicated by (P), frontispieces by (F).

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Out of the Editor's Basket

New Science Hobby Kits

Five new educational kits covering Electronics, Geology, Weather, Optics and Medicine are now being distributed exclusively to the educational and laboratory supply fields by Central Scientific Co., 1700 Irving Park Road., Chicago, Illinois, under an agreement with Industrial America, Inc., coordinators of the hobby kit program.

Each of the kits is prepared by a leading industrial firm in collaboration with the Museum of Science and Industry and Encyclopaedia Britannica. The kits and their producers are: Electronics Kit made by Radio Corporation of America; Optical Kit produced by the American Optical Company; Rock Detective Kit manufactured by the Gemological Institute of America; Weather Kit put together by Taylor Instrument Companies; and the Medical training Kit by Bauer and Black.

Youngsters can learn the hows and whys of each science by building their own scientific instruments from materials contained in the kits. Each kit is also equipped with a comprehensive instructional booklet, prepared under the direction of the Encyclopaedia Britannica. The booklet expalins and illustrates in detail the fundamentals of the science for which the kit was prepared and gives step-by-step directions for the numerous experiments that can be performed.

The five kits now available from Central Scientific Co. are designed for children from 8 to 18 years of age and are actually only the forerunners for what is hoped to be a complete series carrying the junior scientists right into college technical programs. Each of the present kits is labeled with the number 1. The coordinators of the program plan to expand them into more advanced study so that eventually there will be kit numbers 2, 3, 4, and so forth.

For futher information and descriptive literature, write to Central Scientific Company, 1700 Irving Park Road, Chicago, Illinios.

Illuminated Magnifier

E. W. Pike & Co., Inc., has announced complete redesign of the housing for its "Flash-O-Lens" Illuminated Magnifiers.

The newly designed housing has a streamlined, modern appearance. One outstanding feature is a new type of thumbnail-notch grip which facilitates loosening and tightening of the ring that holds the lenses in the housing. Long service life can be expected from the new housing because it is now being molded of durable, heat-resistant polystyrene.

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Further information on Silatube is available from Labtician Products Co., 190–04 99th Avenue, Hollis 23, New York.

Phenylfluorone

A data sheet for phenylfluorone, a sensitive reagent for germanium, has been issued by Jasonols Chemical Corp., 825 East 42nd Street, Brooklyn 10, New York. The sheet contains several references to the literature on phenylfluorone as well as procedure for determination of germanium.

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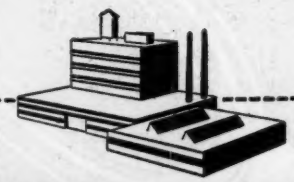

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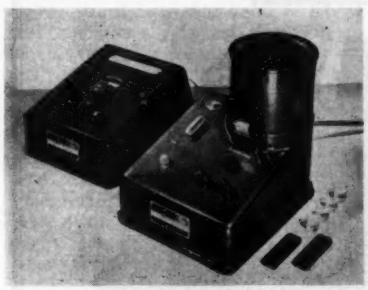

A new photofluorometer, the Coleman Model 12C, is the latest in Coleman Instruments' distinguished line of fluorimetric instruments. The Model 12C offers a greatly increased sensitivity and improved stability which enable it to deliver accurate data both at very faint fluorescence levels and lower concentrations than those previously convenient to handle. Full details are given in Bulletin B-230 which may be had by writing Coleman Instruments, Inc., 318 Madison St., Maywood, Ill.

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A new automatic washer for laboratory glassware and test tubes, the James Laboratory Washer, has recently been introduced by the National Appliance Co., 45 Clinton Place, New Rochelle, N. Y.

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On the "Lift" of a Gas: A Device for Open House

Julius Sumner Miller, of El Camino College, California, contributes the following:

This little business involves some chemistry and some physics, and possesses a goodly measure of the esthetic. Sir William Bragg called it "a beautiful old experiment." In classical days it was done with grapes and champagne but Bragg used soda water instead of champagne! (See "Concerning the Nature of Things" by Sir William Bragg.

Fill a tall glass cylinder with water. Into this drop three or four moth balls. Their composition is naphthalene, whose specific gravity is slightly greater than that of water, and they sink. Now drop a little mossy zinc into the vessel and add some HCl. Hydrogen gas is evolved. This gathers on the surface of the moth balls, whereupon they rise. When they reach the surface they partially emerge, some of the gas escapes, the buoyancy becomes less, and they sink. The process repeats.

If the gas which clings to the moth balls does not provide sufficient "lift" it is interesting and instructive to add a bit of common table salt. The buoyant forces now arising are adequate for upward motion.

When the moth balls hit the free surface, only partially emerging, the gas from the *exposed* surface escapes. The buoyancy on that part still submerged is not uniform

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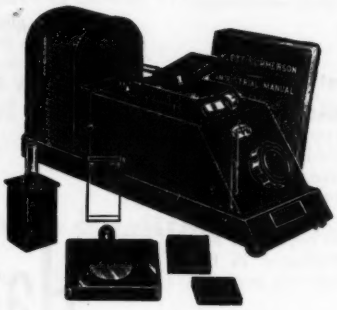

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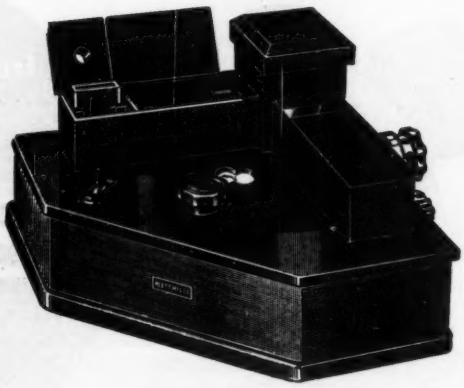

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generally, since the gas is not uniformly gathered, hence the ball turns over. In this process practically all of the gas escapes and the ball sinks. If, now, on its way down one ball strikes another coming up, enough gas may be dislodged so that the one coming up ceases to rise and sinks also.

If a rising ball is carefully observed it will be noted that, as it rises, the bubbles of gas expand. The additional buoyancy thus provided increases the rate of ascent. Further, as the gas gathers on the ball at the bottom of the vessel it will be observed to spin, now this way, now that. These many separate observations lend themselves to good discussion.

Finally, if the liquid is slightly colored (with a pinch of potassium permanganate, say) the whole business has an esthetic attraction. If set up on large scale observers are invited quite irresistibly.

New Literature

● In celebration of the 100th Anniversary of Paul Ehrlich, the current issue of "The Laboratory," published by the Fisher Scientific Co., 711 Forbes St., Pittsburgh 9, Pa., features an interesting, historical sketch about this famous scientist.

● "*Lanco* Apparatus News" for October, Vol. 6, No. 2, features Stuart and Briegleb atom models. This 12-page bulletin describes two dozen other items of laboratory equipment. Among these are apparatus for subsieve range particle size analysis, test sieve vibrator, balance with 311-g. capacity, infrared water bath, and lattice rod support clamps. For copies write to Arthur S. LaPine & Co., 6001 S. Knox Ave., Chicago 29, Ill.

● A. Daigger & Co., Kinzie at Wells, Chicago 10, Ill., has enlarged its publication of the "Apparatus Digest" to 16 pages. Issued several times per year, the Digest details and illustrates a wide scope of apparatus, instruments, and other laboratory materials suitable for testing, research, and control laboratories. Included also are brief editorial articles on scientific developments in general. The Digest is distributed gratis to laboratory personnel in industry, in Federal agencies, colleges, and the medical sciences.

Subjects in the current issue include Vernier action needle valves, ultraviolet black light in science and industry; flexible wrap-around heating tapes; giant dial stopclocks; laboratory engineered notebooks; inexpensive humidity indicating cards; Pyrex brand electrically conductive heating tubes; portable refrigeration systems; and a variety of other devices including pumps, stirrers, heaters, hot plates, and stainless steel laboratory ware. The Digest is available on request.

● Publication of a new series of molybdenum chemical data bulletins was recently announced by the Climax Molybdenum Co. These bulletins are designed to provide, in concise form, authoritative data on the properties of molybdenum compounds. Previously such data were often difficult to find in the literature and hard to evaluate.

The first bulletin in the series is entitled "Properties of Molybdic Oxide." It presents selected values for the physical properties of this basic molybdenum compound and a review of its chemical proper-

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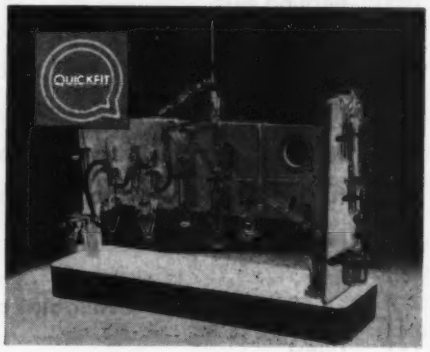

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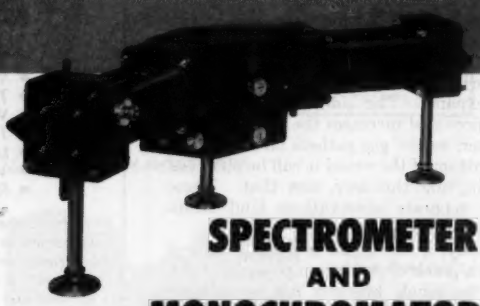

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Science Education, April 1954

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ties. Molybdic oxide is used as a catalyst, an intermediate for the production of molybdates and metallic molybdenum, an alloying agent for steel and a constituent of special ceramics.

New bulletins in this series will appear at intervals of several months. Other titles planned for the next year include, "Thermodynamic Properties of Molybdenum Compounds," "Molybdenum Pentachloride," "Simple Molybdates," "Complex Molybdates" and "Organic Molybdenum Complexes."

Copies of this data series are available upon request to the Climax Molybdenum Co., 500 Fifth Ave., New York 36, N. Y.

New Literature

● The Smithsonian Institution has just published the ninth revised edition of the "Smithsonian Physical Tables," compiled by Dr. W. E. Forsythe. These comprise 901 tables, including completely revised data appearing in previous editions plus new sections on nuclear and atomic physics. This volume should prove especially useful as a reference work for libraries, physicists, scientific laboratories, and science teachers. Orders should be sent to the Distribution Section, Editorial and Publications Division, Smithsonian Institution, Washington 25, D. C. Prices, postpaid: $9, paper bound; $10, cloth bound.

● Armour Research Foundation has recently issued a little booklet on "Solution Ceramics," a new way for industry to produce ceramic coatings which are not brittle and can be applied easily to almost any clean solid surface at a few hundred degrees Fahrenheit. The booklet lists several possible uses for solution ceramics and answers various questions regarding physical properties and attributes of these coatings. Further information about the process is available from E. W. Wickert, Assistant Business Manager, Armour Research Foundation of Illinois Institute of Technology, 10 W. 35th Street, Technology Center, Chicago 16, Illinois.

● A recent publication of the Council of Scientific and Industrial Research of New Delhi, India, is a paper-bound book entitled "Survey of Research Problems in Plastics with Special Reference to the Development of Plastics Industry in India." The book is illustrated with photographs. Some chapter titles are: Fundamental Researches in Plastics, Natural Resins and Resin-like Materials, Naturally Occurring Polymers, Polycondensation Products, Polymers Based on Ethenoid Unsaturation, Processing of Plastics, Raw Materials, etc. Information on the book is available from K. Venkataraman, Assistant Secretary, C. S. I. R. Publications Division, Old Mill Road, New Delhi 2, India.

● Another new paper-bound book is the UNESCO's "Inventories of Apparatus and Materials for Teaching Science. Volume III: Technical Colleges. Part 4: Electrical Engineering." The book lists in detail the teaching of electricity in two specific schools, one in France and one in Sweden, and teaching of electricity in Great Britain in general. Information about the object and character of the

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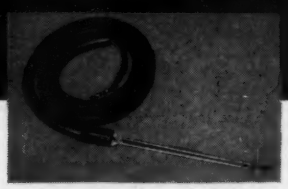

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While burner gives off little heat—no cooling necessary—flame is concentrated to give high temperature. Greatly increased excitation of metallic ions assures increased sensitivity without interference from radiation of organic molecules. The stability and temperature of flame coupled with constant volume atomizing eliminate the use of internal standards such as lithium. Through simple direct-coupled optics of the Model 21, light from flame is focused directly on high vacuum phototube by a single lens . . . no mirrors or prisms to align, no slits to adjust. Avoided is the tremendous electronic amplification required by less efficient systems and resulting maintenance and repair. For isolating radiation of element under analysis, filters are provided with the instrument. These filters are selected and adjusted for the measurement of Sodium, Potassium and Calcium in blood plasma, urine and other solutions.

For detailed information about the New Coleman Model 21 Flame Photometer, write E. Machlett & Son for Bulletin S231.

C76-997 Coleman Flame Photometer for use with any Coleman Spectrophotometer or Photometer and including sodium and potassium filters.....$450.00

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teaching, the organization of the schools, entrance requirements, examinations, the teaching program, materials and equipment, content of specific courses, etc., is given for each. National distributor of UNESCO publications for this country is the Columbia University Press, 2960 Broadway, New York 27, N. Y.

● A 26-page two-color brochure on the safe handling of chlorine has just been issued by the Columbia-Southern Chemical Corporation, wholly-owned subsidiary of Pittsburgh Plate Glass Company.

Subject matter covered in the booklet includes details on methods of shipment, instructions for handling, storage, removal from, care, and return of containers, etc. Complete information on methods to use in tracing leaks as well as measures to be employed in coping with them is covered.

A detailed section on first-aid measures is included in the booklet, which is illustrated throughout. The "Chlorine Safe Handling" brochure may be had free on request to Columbia-Southern, 632 Fort Duquesne Blvd., Pittsburgh 22, Pa.

● A new Kodak Data Book, "Processing Chemicals and Formulas," which combines the information in the former editions of the Data Books, "Processing and Formulas," and "Chemical Preparations," has just been issued by the Eastman Kodak Company.

Data formerly presented have been completely revised and enlarged so that the new Data Book will provide the latest information on processing, preparation of solutions, formulas, and descriptions of Kodak prepared chemicals. The purpose of this information is not only to provide the reader with instructions, but also to explain briefly the basic elements of the operation and the reasons why certain techniques produce desired effects. One interesting detail is the inclusion of comparison color patches for use with the test for thoroughness of washing of photographic prints.

This new Data Book contains 68 pages, and is punched for binding in the Kodak Photographic Notebook or Reference Handbook. It costs 50 cents per copy and is available through Kodak dealers.

● Sargent's latest issue of Scientific Apparatus and Methods, Vol. VI, Number 3, is now available. An illustrated 40-page magazine-catalogue, it features the new Sargent Thermistor Thermometer, a variable multi-range indicating instrument used in temperature measurements. Also fully described is an oscillometric analysis of two-component single phase liquids. The catalogue supplement section contains four types of Sargent Thermonitors, thermonitor-controlled water and viscosimeter baths, as well as 32 additional pieces of laboratory instruments, equipment, and glassware. A copy may be obtained by addressing: The Advertising Dept., E. H. Sargent & Co., 4647 W. Foster Ave., Chicago 30, Illinois.

● Available now without charge is the newest edition of the Fisher Gram-atic Balances bulletin, an eight-page, fully-illustrated work that gives the theory and operation of single-pan balances.

This new edition has a section on the

(*Continued on page 36*)

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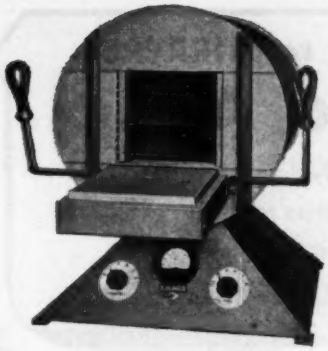

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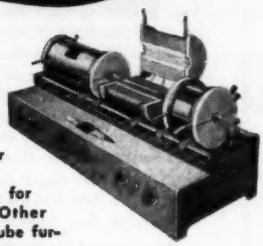
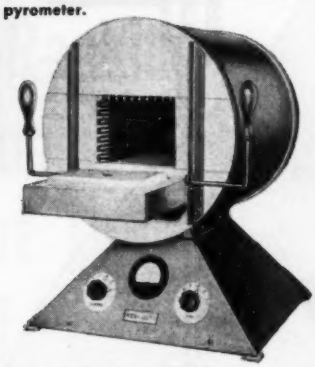


Three separately controlled tube furnaces mounted on slide rails, for use individually or as one assembly. Hinged construction for easy operation. Other sizes and types of tube furnaces available for temperatures to 2600° F.



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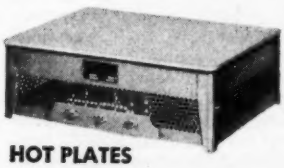

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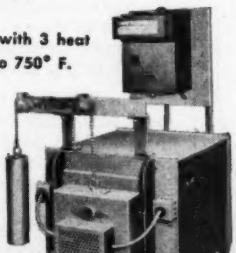

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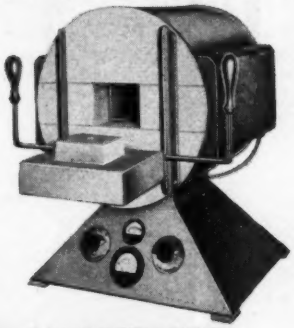

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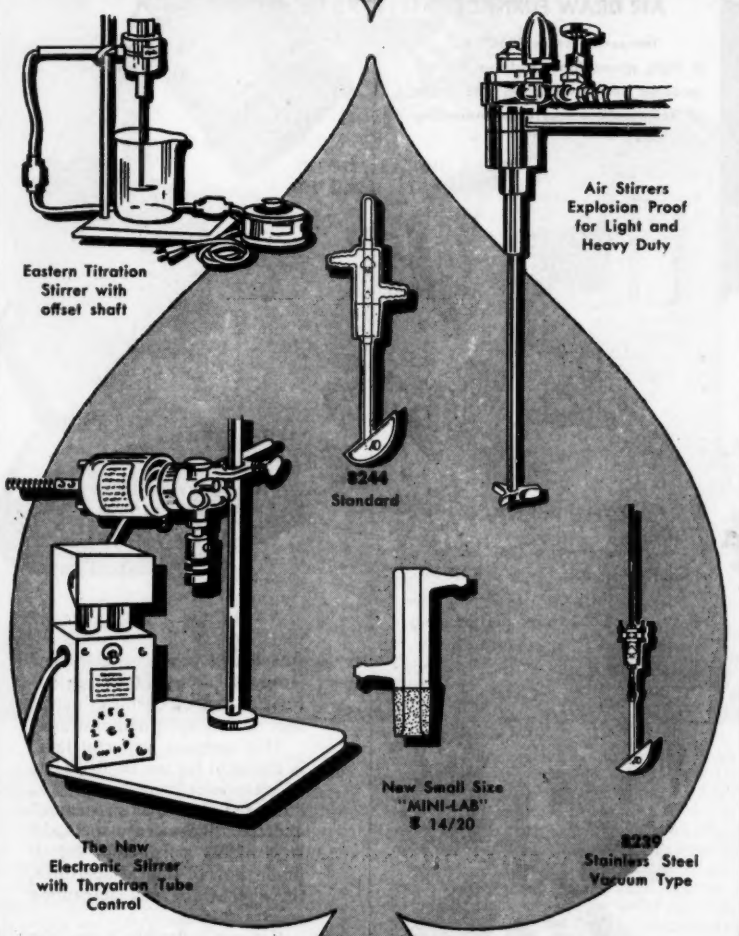
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VINELAND NEW JERSEY

(*Continued from page 34*)

Micro Gram-atic Balance as well, a single-pan constant-sensitivity balance with an accuracy of ±0.002 milligrams, said to require one-third to one-quarter the time of conventional micro balances. Also included in the bulletin is a description of a special-purpose single-pan balance for weighing large materials that will not fit inside regular weighing compartments.

Bulletin number is FS-207, and it is available from Fisher Scientific Co., 717 Forbes St., Pittsburgh 19, Pennsylvania.

● Two new catalogues published by Consolidated Engineering Corp., 300 N. Sierra Madre Villa, Pasadena, 8, Calif. are now available. Bulletin CEC 1302 A is entitled "Analytical and Control Instruments" and graphically illustrates information on analytical, isotope-ratio, dual-purpose and process-monitoring mass spectrometers; high vacuum leak detectors; Titrilog sulfur recorders, etc.

Bulletin CEC 1301 is entitled "Data Processing Instruments" and describes oscillographs; galvanometers; amplifiers; velocity pickups; oscillogram processors; vibration meters analog-to-digital converters and power supplies.

● Pacific Transducer Corp. offers, without charge, a new circular and calibration chart. This shows conversions for latex foam rubbers, sponge rubbers and rubber-like plastic sponge for the various scales of the American Society for Testing Materials, Rubber Manufacturers Association, Scott and Shore A scales and the Pandux scale. According to Pacific Transducer, this is the first time these various scales have been correlated so that one can convert any one scale to another at a glance. Copies of the chart may be secured by writing to the company at 11836 West Pico Blvd., Los Angeles 46, California.

● The 1955 Denver Annual Diary, compiled by Denver Equipment Company to provide an efficient device for keeping permanent records of daily expenses, investments, insurance, taxes, vacations, birthdays, addresses, and phone numbers, is now available. A convenient size for handling and filing, the diary is a simple but excellent record of facts and figures so necessary for income tax forms. Business and personal records are made readily available in the Denver Diary. For your copy write to Denver Equipment Company, P.O. Box 5268, Denver 17, Colorado.

● "Put Wheels to Work in Your Laboratory" is the title of a new 12-page illustrated folder published by Laboratory Construction Company. The folder describes special purpose carts and portable tables. It shows at a glance the variety of uses carts serve in research, industrial, medical, and educational laboratories. It demonstrates how this equipment speeds laboratory work, reduces breakage of glassware and instruments, helps cut labor costs and adds mobile work space. Besides large photographs of the carts in use, the new folder contains diagrams, descriptions, and prices. Included are glassware carts, chemical carts, flask carriers, and portable instrument tables. For a free copy of the folder, write Laboratory Construction Company, 1115 Holmes Street, Kansas City, Missouri.

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Assuring a high degree of accuracy in the instrument is the precision ground glass plunger. To provide for a vacuum tight seal the plunger is sealed in with a Teflon gasket, backed up with silicone rubber.

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G 15395C Micro Pipet-Buret, Gilmont, Combination for 1 ml. and 0.1 ml. Capacities. **ea. 82.50**

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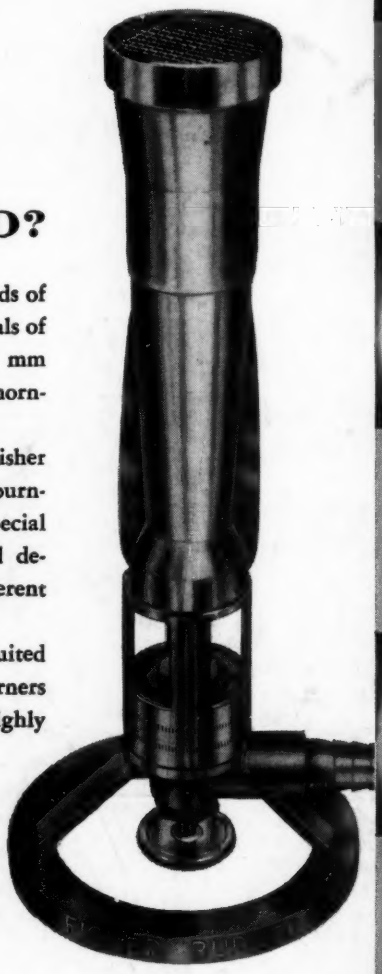

Gas is used in the laboratory for all kinds of heating, from micro-fusions of a few crystals of unknown precipitate to U-bends in 25 mm tubing. (And let's not forget the beaker of morning coffee!)

For general purposes, the patented Fisher Burner is the most useful and efficient gas-burning unit the laboratory can have, but for special applications the Fisher Catalog lists and describes *forty-seven* burners, each with different characteristics and special features.

Why not select the apparatus that's best suited for your individual needs? Whether it's burners or thermometers, spatulas, stoppers or highly specialized instruments . . .

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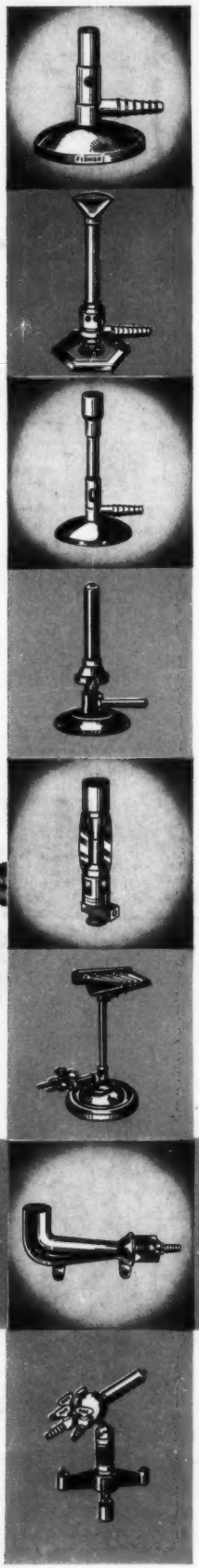

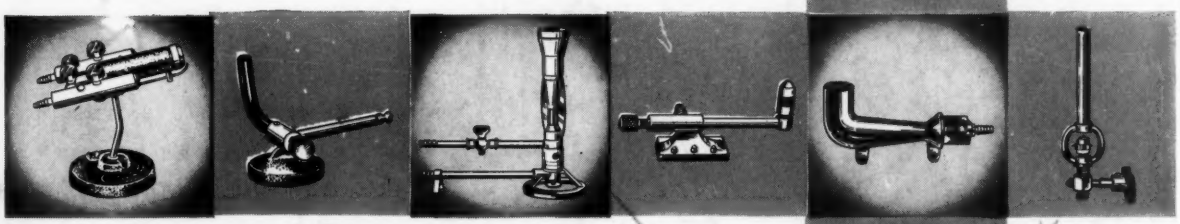

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